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Canada Cement Lafarge Ltd.

Energy Conservation in the Industrial Minerals Processing Sector Comminution (Crushing and Grinding) Phase III

Activity 3

Chemical Additives – Their Current and Potential Uses in the Dry Grinding of Non-Metallic Minerals

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ACTIVITY 3

Chemical Additives - Their Current and Potential Uses in the Dry Grinding of Non-Metallic Minerals

WITTECK DEVELOPMENT INC.
2640 South Sheridan Way, Mississauga, Ontario, Canada L5J 2M4
Telephone: (416) 823-7381 Fax: (416) 823-5689

ACTIVITY 3
CHEMICAL ADDITIVES - THEIR CURRENT AND POTENTIAL USES IN THE DRY GRINDING OF NON-METALLIC MINERALS

ENERGY CONSERVATION
IN THE INDUSTRIAL MINERALS PROCESSING SECTOR
COMMINUTION (CRUSHING AND GRINDINC) PHASE III
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LAFARGE CANADA INC.
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H4P 2R3

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J. Kriens
President, IMD Laboratories

Louis M. Bernard, P. Eng
General Operations Manager

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1.0 EXECUTIVE SUMMARY

The Department of Energy, Mines and Resources'(E.M.R.) Canada Centre for Mineral and Energy Technolgy (CANMET) in their wish to assist the industrial minerals sector in improving their energy efficiency and to promote further research commissioned a study on the uses and benefits of chemical additives in dry comminution.

- The overall objective as stated by EMR was to conduct a literature survey of internationally published literature on the research, development, and applications of chemical additives in the dry grinding of cement and other industrial minerals.

- To prepare a detailed bibliography of published literature.

- To design a questionnaire directed to a broad range of industrial mineral and cement processing companies, with the objective of establishing the degree of use of chemical additives in comminution and the overall effect on energy savings and/or process efficiencies.

- To conduct bench scale static grinding tests, using chemical additives of various types as suggested by other researchers on the following materials: cement, calcium carbonate, silica, nepheline syenite and talc.

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1.1 Study Objectives

A detailed study on the uses and potential benefits of the chemical additives in the dry grinding of industrial minerals and cements was conducted by I.M.D. Laboratories Limited. The study was broken down into several phases.

- To conduct a detailed computer assisted literature search, on data published by various researchers and others in scientific journals, patents, seminars and conferences.

- The preparation of a questionnaire, of which one part was specifically directed to the cement industry and the second part to industrial minerals processors.

- Distribution of the questionnaires, followed by analyzing the responses received.

- Conducting bench scale "static grinding" test using a number of various grinding aids for dry grinding of cement, calcium carbonate, silica, nepheline syenite and talc.

1.2 Description of Study Phases

1.2.1 Literature Search

A literature search was conducted to obtain pertinent published literature dealing with the subject of the use of chemical additives in the dry grinding of solids, in particular non-metallic minerals and cement. For the purpose of studying their effectiveness in improving the dry grinding process or enhancing solid flow, dry dispersion and air classification, the search was conducted via computer using the data bank of the Canadian Institue for Scientific and Technical Information (C.I.S.T.I.) of the National Research Council in Ottawa, Canada.

1.2.2 Questionnaires

The questionnaires were prepared to:

- o determine the extent of chemical additives or grinding aids used in grinding of cements and other non metallic minerals;
- o to evaluate the benefits derived from their use, with respect to energy conservation, process efficiency, product quality and performance;
- o to obtain information on the type of chemical additives and method of implementation used in the grinding process.

Seventy-five percent of the questionnaires were mailed to North American producers of cement and other industrial minerals. Twenty-five percent were sent to European and South American producers.

A total of one hundred and forty six (146) questionnaires were sent out, of which one hundred and one (101) were directed at the cement industry and forty five (45) to various industrial mineral producers. A total of forty six (46) valid responses were received of which thirty five (35) were from cement producers and eleven (11) from industrial minerals producers. The response was thirty-five (35) percent from the cement industry and twenty-four (24) percent from industrial mineral producers.

The responses indicated extensive utilization of chemical additives in the cement industry, with improvements in productivity and energy consumption of up to twenty five (25) percent.

1.2.3 Bench Scale Grinding Tests

From data reported in published literature and from responses to the questionnaires, a group of chemical additives were selected for evaluation in the grinding of cement and other selected minerals.

The testing was conducted with materials supplied by various industrial mineral producers and a Canadian cement company.

The tests were conducted using a conventional ceramic laboratory jar grinding mill charged with fused alumina grinding media.

A direct comparison was made between materials ground with and without selected chemical additives.

1.3 Concept Of Using Chemical Additives In The Comminution Of Industrial Minerals And Cement

It is well documented that in excess of 97% of the energy consumed in mineral grinding is wasted as heat. Only 0.5 to 1.0 % is creating new surfaces.

Grinding naturally reduces particle size. As particles are reduced in size, the surface area of the ground material increases and continued grinding becomes progressively more difficult requiring ever increasing amounts of energy to achieve further size reduction. Eventually, a practical and economical limit of grinding process is reached.

One important factor limiting size reduction is the tendency of fine particles produced during the grinding process to re-aggregate : i.e. form soft agglomerates. An equilibrium is established between two distinctly different processes: i.e. agglomeration and fragmentation.

There is no absolute clear understanding of this phenomena other than that it is caused by various types of physio-chemical forces; such as Van der Waals charges, electrostatic forces, plastic deformation by fracturing and/or breaking the crystal structure. The aggregation exhibits itself as a balling up, as if the material is damp: i.e. formation of soft agglomerates. These aggregates often consist of a coarser particle coated with many fine particles. This stage of aggregation is due to Van der Waals forces and can be mechanically reversed since the energy involved is only 0.04 to 4 KJ/mole. The secondary agglomerative phase which develops upon further grinding results in the formation of hard agglomerates or caking resulting from energy levels as high as 40 to 400 KJ/mole, and cannot be reversed mechanically.

The agglomerative phase is accompanied by severe coating of grinding media and mill shell, with a hard eggshell like coating that continues to build up. These coatings cushioning effect prevents the comminution process to proceed in its normal manner.

These phenomena also contribute to a number of secondary problems, such as poor flow characteristics through the grinding mill: i.e. an artificially created increase in residence time; plugging of discharge grates; and in the case of grinding systems coupled with air classifiers, in poor classification efficiency due to lack of dispersion of the material to be classified.

Air classification, in fact, can compound the problems as the classifier will reject the aggregates, agglomerates or flakes as if they are coarse particles, returning them to the grinding mill for further grinding.

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The problems illustrated above will be most noticeable in operations where it is necessary to fine grind to a very small particle size. They may occur at different levels of particle size for different minerals or even of the same mineral from a different geological source.

Other factors such as mill residence time, size of grinding media, type of mill discharge or the open area of the discharge grate can compound these problems.

The function of chemical additives are to eliminate or restrict these problems from developing, or at least minimize their effect. When effective, chemical additives will result in increasing production at equal particle size and kWh energy input, or in producing an equal amount of materials but of finer particle size at the same energy level. Therefore, industrial minerals and cement produced can benefit from the use of chemical additives.

Before using them, a test program should always be conducted in the laboratory to evaluate their potential performance and benefit.

The tests were conducted with the objective to establish the following:

- The rate of grinding: i.e. the amount of material finer than 325 mesh (45 micron) produced in a specific time period for each material ground with and without additives.

- The behaviour of the ground material with respect to formation of aggregates or agglomerates and observations on ball and mill wall coating.

- Analyzing various chemical additives with respect to their effectiveness in improving the efficiency of the comminution process.

- The purpose of the testing was to investigate the potential of chemical additives to:

1. Grind a given amount of a mineral or cement to a specific size or surface area in a shorter time period compared to the same amount of the mineral or cement ground without an additive.

2. Determine to what extent chemical additives will retard or prevent agglomeration or coating of grinding media and mill walls.

Table 1.3.1 summarizes results of tests on cement and the other selected minerals with the most promising chemical additives.

Detailed results from the bench scale testwork are given in the body of this report.

1.4 Conclusions

- Tri-ethanolamine was the most effective grinding aid tested for cement, calcium carbonate and nepheline syenite. Detailed test results are shown in tabular/graphical form in the respective sections of the report.

- The review of published literature gave a strong indication that the use of chemical additives are widespread in the cement industry.

- Questionaire responses industrial mineral producers indicates that chemical additives have not received the same degree of attention.

Table 1.3.1

SUMMARY OF BEST TEST RESULTS ON BENCH SCALE TEST

Material	Grinding Aid	Grinding Time (In Hours)	% Retained On 45 Micron	Blaine Surface Area cm^2/gram
Cement	none	3.5	8.3	2888
	Tri-ethanolamine	2.0	1.7	3081
Calcium Carbonate	none	1.5	11.2	-
	Tri-ethanolamine	1.5	1.4	-
Silica (Quartzite)	none	2.5	12.6	-
	Silicone-glycol	2.5	4.6	-
Nepheline Syenite	none	2.5	15.8	-
	Tri-ethanolamine	2.5	2.8	-
Talc	No Effective Grinding Aids Were Found			

- It is the authors opinion that the use of chemical additives, particularly in the grinding of calcium carbonate is more extensive than is indicated by the responses to the questionnaire.

- Potential benefits resulting from the use of chemical additives are well documented in the published literature. Selection and method of use of chemical additives must be based on detailed laboratory testing.

- Implementing the use of chemical additives must consider other pertinent factors in the grinding classification process such as the effect chemical additives may have on the industrial applications of products ground with chemical additives.

- Results depend not only on the chemical additive but also on the optimum operation of the grinding system itself. Changes in mill retention, total mass flow in the system could require modifications to operating procedures and/or modifications to the actual grinding equipment such as discharge grates, grinding media size and material handling systems.

This is discussed in detail in the body of this report.

- Despite a wealth of scientific data, there still is no absolute agreement, understanding or well stated theory that clearly establishes the mode of operation of chemical additives.

- Interrelated factors, such as mineral origin, mineralogical composition and equipment variables tend to cause difficulty in precise interpretation of test results obtained by various researchers. In some instances a chemical additive found to be effective on a given material under certain milling conditions was found to be ineffective on a similar material from a different deposit or under different grinding conditions.

- This illustrates that it is necessary to conduct laboratory tests to screen various potential chemical additives. Once a promising additive has been found by laboratory testing, its successful implementation may require a change in grinding conditions or changes to grinding equipment such as media size, grate discharge opening or levels of circulating loads.

1.0 RÉSUMÉ DU PROGRAMME DIRECTEUR

Désireux d'aider le secteur des minéraux industriels à améliorer le rendement énergétique de ses produits et de favoriser l'avancement de la recherche, le Centre canadien de la technologie des minéraux et de l'énergie (CANMET) du ministère Énergie, Mines et Ressources Canada (EMRC) a commandé une étude sur les utilisations et avantages des additifs chimiques dans la fragmentation à sec.

L'objectif global formulé par EMRC était le suivant:

- Faire une recherche bibliographique sur la recherche, le développement et les applications concernant les additifs chimiques dans le broyage à sec du ciment et d'autres minéraux industriels, au niveau international.

- Dresser une bibliographie détaillée de toute la documentation publiée.

- Préparer un questionnaire destiné à un grand nombre de compagnies de traitement de ciment et de minéraux industriels afin d'établir le degré d'utilisation d'additifs chimiques dans la fragmentation et d'en évaluer les retombées en termes d'économies d'énergie et d'efficacité au niveau des procédés.

- Effectuer des essais de broyage statique en laboratoire, c'est-à-dire tester les divers types d'additifs chimiques suggérés par les chercheurs sur les matériaux suivants: ciment, carbonate de calcium, silice, syénite néphélinique et talc.

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1.1 Objectifs de l'étude

Une étude détaillée portant sur les utilisations et les avantages possibles des additifs chimiques dans le broyage à sec des minéraux industriels et des ciments a été menée par I.M.D. Laboratories Limited. L'étude comprenait quatre étapes:

- Effectuer une recherche assistée par ordinateur dans le but d'établir une liste détaillée des articles publiés par les chercheurs et autres spécialistes dans des revues scientifiques de même que des brevets, séminaires et conférences.

- Préparer un questionnaire en deux volets, le premier réservé à l'industrie du ciment et le second, aux entreprises spécialisées dans le traitement des minéraux industriels.

- Distribuer le questionnaire, puis analyser les réponses reçues.

- Faire des essais de broyage statique en laboratoire dans le but d'étudier le comportement d'un certain nombre d'aides dans le broyage à sec du ciment, du carbonate de calcium, de la silice, de la syénite néphélinique et du talc.

1.2 Description des étapes de l'étude

1.2.1 Relevé des publications

Une recherche a été effectuée par un ordinateur explorant la banque de données de l'Institut canadien de l'information scientifique et technique du Conseil national de recherches Canada, Ottawa, dans le but, premièrement, de repérer toute la documentation portant sur l'utilisation d'additifs chimiques dans le broyage à sec de matières solides, notamment les minéraux non métalliques et le ciment et, secondement, de vérifier l'aptitude de ces aides de broyage à améliorer le broyage à sec ou à favoriser un meilleur débit des matières solides, une meilleure défloculation à sec et une meilleure classification à l'air.

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1.2.2 Questionnaire:

Le questionnaire a été préparé aux fins suivantes:

- o déterminer l'étendue de l'usage d'additifs chimiques ou d'autres aides dans le broyage du ciment et de minéraux non métalliques;

- o évaluer les avantages découlant de leur utilisation en termes de conservation d'énergie, d'efficacité au niveau du traitement, de la qualité du produit et du rendement;

- o obtenir de l'information sur les types d'additifs chimiques et sur les méthodes utilisées dans le procédé de broyage.

Soixante-quinze pour cent des questionnaires ont été postés à des producteurs nord-américains de ciment et d'autres minéraux industriels, tandis que vingt-cinq pour cent ont été envoyés à des producteurs européens et sud-américains.

Un total de cent quarante-six (146) questionnaires ont été distribués. De ce nombre, cent un (101) étaient destinés à l'industrie du ciment et quarante-cinq (45) à divers producteurs de minéraux industriels. En tout, on a reçu quarante-six (46) réponses admissibles, dont trente-cinq (35) provenaient de producteurs de ciment et onze (11) de producteurs de minéraux industriels. Le taux de réponse a été de trente-cinq (35) pour cent dans le cas de l'industrie du ciment et de vingt-quatre (24) pour cent dans le cas des producteurs de minéraux industriels.

L'analyse des réponses a permis d'établir que l'industrie du ciment fait une utilisation massive d'additifs chimiques et que ces derniers permettent d'améliorer la productivité et de diminuer la consommation d'énergie jusqu'à concurrence de vingt-cinq (25) pour cent.

1.2.3 Essais de broyage en laboratoire

À la lumière des données tirées de la documentation publiée et des réponses au questionnaire, on a décidé d'évaluer le comportement d'un groupe d'additifs chimiques dans le broyage du ciment et d'autres minéraux sélectionnés.

Les essais ont porté sur des matériaux fournis par différents producteurs de minéraux industriels et par une compagnie de ciment canadienne.

Les essais ont été effectués dans un broyeur de laboratoire classique de type à contenant en céramique rempli d'alumine fondue agissant comme corps broyant.

Ensuite, on a fait une comparaison directe entre les matériaux broyés avec et sans les additifs chimiques sélectionnés.

1.3 L'utilisation d'additifs chimiques dans la fragmentation des minéraux industriels et du ciment

Il a été démontré que plus de 97% de l'énergie consommée pendant le broyage des minéraux se perd sous forme de chaleur. Seulement 0,5 à 1,0% de l'énergie sert à créer de nouvelles surfaces.

Le broyage a pour but de réduire la grosseur des granules. À mesure que les particules diminuent en taille, la surface du matériau broyé augmente, ce qui a pour effet d'ajouter progressivement à la difficulté du broyage et à la consommation d'énergie. Éventuellement, on en arrive à atteindre une limite technique et économique du procédé de broyage.

Comme les fines particules produites en cours de broyage ont tendance à s'agglomérer, c'est-à-dire à former de nouveaux agrégats mous, elles limitent la possibilité de réduire davantage la grosseur des granules. Un équilibre est atteint entre deux procédés tout à fait distincts: l'agglomération et la fragmentation.

Ce phénomène n'est pas parfaitement compris, bien que l'on sache qu'il résulte de forces physiochimiques comme les charges de Van der Waals, les forces électrostatiques et la déformation plastique causée par le fractionnement et le bris de la structure cristalline. Cette agrégation se manifeste par la formation de balles, comme si les matériaux étaient humides, d'ou la notion d'agglomérats mous. Ces agrégats sont souvent constitués de particules grossières recouvertes de particules fines. Comme la première phase de l'agrégation est due aux forces de Van der Waals, elle peut être mécaniquement renversée puisque l'énergie en jeu n'est que de 0,04 à 4 KJ/mole. Quant à la seconde phase, qui se développe par suite d'un broyage soutenu, elle entraîne la formation d'agglomérats durs ou l'agglutination (croûte). Comme ce dernier phénomène est lié à une consommation d'énergie aussi élevée que de 40 à 400 KJ/mole, il ne peut être renversé mécaniquement.

Cette seconde phase s'accompagne d'une importante agglomération de particules autour des corps broyants et de la virole du broyeur; cette couche, dont la dureté est comparable à celle d'une coquille d'oeuf, ne cesse de grossir ou d'épaissir, en plus d'empêcher la fragmentation de se faire normalement en raison de l'effet amortisseur de ce nouveau revêtement autour des corps broyants.

Ces phénomènes entraînent également un certain nombre d'effets secondaires comme le mauvais écoulement des granules dans le broyeur - ce qui augmente artificiellement le temps de traitement - l'obstruction des grilles de décharge et, dans le cas de systèmes de broyage comprenant des classificateurs à air, une mauvaise classification en raison du manque de dispersion des matières à classifier.

De fait, la classification à l'air peut ajouter au problème puisque le classificateur rejettera les agrégats et les agglomérats ou floçons au titre de particules grossières ou trop grosses, puis les recyclera aux fins de traitement supplémentaire.

De plus, les problèmes susmentionnés ne font que s'accentuer lorsque l'on est appelé à procéder à un broyage fin des particules, c'est-à-dire les réduire à des dimensions extrêmement petites. Ils peuvent survenir avec différents minéraux de grosseurs de particules différentes, ou encore avec des minéraux identiques provenant de sources géologiques distinctes.

D'autres facteurs comme le temps de séjour dans le broyeur, la taille des corps broyants, le type de décharge et la section libre ou l'aire ouverte de la grille de décharge peuvent ajouter à ces problèmes.

Le rôle des additifs chimiques consiste à éliminer ou à empêcher ces problèmes de se manifester, sinon de réduire leurs effets au minimum. Lorsqu'ils remplissent leur fonction, les additifs chimiques augmentent la production à grosseur de particules et à nombre de kilowattheures égaux, ou encore produisent une quantité égale de matériaux, mais d'une plus grande finesse, à dépense énergétique comparable. Résultat: l'utilisation d'additifs chimiques est avantageux dans la division des minéraux industriels et du ciment.

Cependant, avant de les employer, il convient de toujours procéder à des essais en laboratoire dans le but d'en évaluer le rendement et les avantages possibles.

Aussi, des essais ont été réalisés dans le but de déterminer:

- Le taux de broyage, c'est-à-dire la quantité de matériau d'une finesse inférieure à 45 microns (maille de broyage 325) que l'on pouvait obtenir dans un temps spécifique à partir de chaque matériau broyé avec et sans additifs.

- Le comportement des matériaux broyés en égard à la formation d'agrégats ou d'agglomérats, à la formation de balles et à la formation de couches sur les parois du broyeur.

- L'analyse des divers additifs chimiques quant à leur valeur au niveau de l'amélioration de la fragmentation des matériaux.

- Le but des essais consistait à évaluer l'aptitude des additifs chimiques à:

1. Broyer une quantité donnée d'un minéral ou d'un ciment à un degré de finesse préétabli dans un laps de temps plus court que celui que n'aurait exigé le traitement d'une même quantité d'un mineral ou de ciment sans additifs.

2. Déterminer dans quelle mesure les additifs chimiques retardent ou empêchent l'agglomération de particules autour des corps broyants ou sur les parois du broyeur.

Le tableau 1.3.1 donne un aperçu des résultats d'essais réalisés sur le ciment et d'autres minéraux choisis avec les additifs chimiques les plus prometteurs.

Les résultats des essais en laboratoire font partie intégrante de ce rapport.

1.4 Conclusions

- La triethanolamine s'est révélée l'additif chimique le plus efficace dans le cas du ciment, du carbonate de calcium et de la syénite néphélinique. Le détail des résultats d'essais est donné sous forme de graphiques et de tableaux dans les sections pertinentes du rapport.

- L'analyse de la documentation publiée indique clairement que l'utilisation d'additifs chimiques constitue une pratique fort répandue dans l'industrie du ciment.

- D'après les réponses au questionnaire, il appert que les producteurs n'accordent pas tous la même importance aux additifs chimiques.

Tableau 1.3.1

APERÇU DES MEILLEURS RÉSULTATS D'ESSAIS EN LABORATOIRE

Matériaux	Additifs chimiques	Temps de broyage (en heures)	% de rétention 45 microns	Surface Blaine cm^2 de surface/ gramme
Ciment	Aucun	3,5	8,3	2 888
	Triéthanolamine	2,0	1,7	3 081
Carbonate de calcium	Aucune	1,5	11,2	-
	Triéthanolamine	1,5	1,4	-
Silice (quartsite)	Aucune	2,5	12,6	-
	Silicone-glycol	2,5	4,6	-
Syénite néphélinique	Aucun	2,5	15,8	-
	Triéthanolamine	2,5	2,8	-
Talc	Aucun additif chimique efficace n'a été trouvé.			

- Les auteurs sont d'avis que l'utilisation d'additifs chimiques, notamment dans le cas du broyage du carbonate de calcium, est plus répandue que ne permet de le croire l'analyse des réponses au questionnaire.

- Les avantages possibles liés à l'utilisation d'additifs chimiques sont fort bien documentés dans les articles publiés. Le choix de la méthode et des additifs chimiques doit être fondé sur les résultats d'une batterie d'essais en laboratoire.

- L'adoption d'additifs chimiques doit également tenir compte de certains facteurs pertinents au niveau de la classification, par exemple l'incidence des additifs chimiques dans les applications industrielles de broyage à l'aide d'additifs chimiques.

- Les résultats dépendent non seulement des additifs chimiques, mais également de l'exploitation optimum du système de broyage. Toute modification apportée au temps de séjour dans le broyeur ou au débit massique peut obliger à faire des changements aux méthodes et matériel de broyage comme les grilles de décharge, la finesse ou la grosseur des corps broyants et les systèmes de manutention des matériaux.

Ce point est traité en détail dans le rapport.

- Malgré les nombreuses données scientifiques, il ne ressort aucune entente ni théorie formelles établissant clairement le comportement des additifs chimiques.

- L'interrelation de facteurs comme l'origine des minéraux, la composition minéralogique et le type de matériel ajoutent également à la difficulté d'interpréter avec précision les résultats d'essais obtenus par différents chercheurs. En effet, on rapporte des cas où un additif chimique qui s'était révélé efficace pour un matériau donné dans certaines conditions de broyage, s'est avéré inefficace en présence d'un matériau analogue provenant d'un autre gisement ou broyé dans des conditions différentes.

- Tous ces éléments indiquent clairement qu'il importe de procéder à des essais complets en laboratoire avant de sélectionner les additifs chimiques appropriés. Une fois que l'on a arrêté son choix sur un additif qui semble prometteur, il peut être nécessaire de modifier le processus ou le matériel de broyage comme la grosseur des corps broyants, l'ouverture de la grille de décharge, le débit, etc.

2.0 INTRODUCTION

The Canadian Industry Program for Energy Conservation (CIPEC) reported a 15% reduction in energy consumption per tonne of output in 1982, compared to the base year 1974. Much of this saving could be attributed to improved housekeeping. Achieving further conservation would require process modificaton and fuel switching; both require Research and Development (R & D). The areas for work were grouped as follows:

General R & D - applicable to industrial mineral processing in general

Cement R & D - processing of Portland and other hydraulic cements and gypsum plaster,

Clay Products R & D - applicable to the manufacturing of brick, tile pipe, etc.

The Department of Energy, Mines and Resources (EMR) CANMET, wishing to assist the industrial minerals processing sector in improving the energy efficiency of its operations, commissioned this study.

The overall objective as stated by EMR was,

> To develop the necessary technology to optimize the comminution of industrial minerals in terms of direct and indirect energy consumption.

EMR defined direct and indirect energy consumption in the following way:

> Direct energy consumption measures the work done during the crushing and grinding operations and is expressed in kWh/tonne.
>
> The indirect energy consumption is less frequently recognized and harder to quantify. Its effects are felt during secondary processing or utilization of comminuted materials. The correct size distribution of cement for example could command savings of up to 10% of the amount of cement necessary to make concrete. Optimization of comminution both in terms of kWh/tonne and in terms of processing energy consumption would make a major contributon to energy conservation in the industrial minerals processing sector.

Ten (10) industrial minerals were chosen for study and, to achieve the objective, the work was divided by EMR into three phases.

Phase I Plant Studies To Develop a Data Base

For each mineral, studies were made of power consumption during comminution at a plant whose operations are typical of the industry. The results are from a brief investigation of the company's operations and therefore, are descriptive rather than analytical.

Phase II Establishment Of Monitoring Methodology

An evaluation was made of commercially available particle size measuring equipment with a view to establishing the feasibility of mill circuit control by on-line particle size measurements.

Also, under this phase, a proposal for a pilot plant test program was developed.

Phase III Pilot Plant Studies of Comminution Related To Industrial Minerals And Energy Consumption

This report is presented in accordance with Phase III, Activity 3 of the study on "Energy Conservation in the Industrial Mineral Sector - Comminution" for Canada Centre for Mineral and Energy Technology (CANMET), Energy, Mines and Resources Canada (EMR).

Witteck Development Inc. of Mississauga, Ontario, and I.M.D. Laboratories Limited of Toronto, are responsible for Activities 1, 2, 3, 4, 6 and 7 of the Phase III programme. The activities include the following:

Activity 1 Examination of the performance of a "state of the art" high efficiency classifier in a conventional milling circuit.

Activity 2 Examination, study and analysis of the state of development of classifiers for cement grinding circuits.

Activity 3 Examination of the use of grinding aids and anti-static agents in dry grinding circuits.

Activity 4 Monitoring and control of a dry grinding circuit using a particle size analyzer and programmable controllers.

Activity 6 Evaluation of the use of dry autogenous grinding of silica to produce coarse size sand grade product.

Activity 7 Development of the necessary technology to optimize the comminution of mica.

The Activity 3 report presents the results of work conducted on the use of chemical additives in the dry grinding of selected industrial minerals and cement at various levels of detail. The study was conducted by I.M.D. Laboratories Limited.

The report contains the following:

- An Executive Summary which outlines the objectives of the study and test program; provides a short discussion of the study phases; and gives a general presentation of the concepts of using chemical additives in dry grinding of industrial minerals and cement. Its role is to provide a general understanding of the use of chemical additives and to highlight results achieved in the testwork.

- A detailed discussion on the findings of other researchers in the use and effectiveness of chemical additives.

- Results of actual bench scale grinding tests, evaluating a number of different chemical additives in the dry grinding of selected minerals and cement.

- A bibliography of reports and articles on the uses and effectiveness of chemical additives, for those wishing to conduct further research and development in this field of endeavour.

- Results from the questionnaire prepared to determine the industrial use of grinding aids.

- Working papers resulting from the above activities which are available for consultation.

3.0 OBJECTIVES

This report examines the technology of applying chemical additives in assisting the dry grinding of industrial minerals and cement. The primary objective is to determine if chemical additives can improve the "Energy Effectiveness" of the comminution process.

To establish the general objectives the following activities had to be undertaken.

3.1 Conduct a detailed literature search of published articles, patents and other pertinent information.

3.2 To review literature as obtained with particular emphasis on the use of chemical additives in the dry grinding of industrial minerals and cement.

3.3 To prepare a detailed bibliography of articles and patents in order to assist others in persuing this particular area of research and development or to provide guidance to companies wishing to investigate if chemical additives can assist in improving the comminution process in their respective grinding operations.

3.4 To prepare detailed questionnaires, directed to cement and industrial minerals producers with the objective of determining the following:

3.4.1 To what extent are chemical additives used by the cement and industrial minerals producers.

3.4.2 Have chemical additives been effective in reducing energy consumption per tonne of product produced. What other benefits have been observed. Which grinding additive is used and at what rate. In addition, the questionnaire sought to obtain information on the type of grinding and air classification equipment in use. The completed questionnaires were returned to the Canada Centre for Mineral and Energy Technology (CANMET). To protect the confidentiality of the information completed questionnaires were sent to I.M.D. Laboratories with the names of the responding companies removed.

3.5 To conduct bench scale static grinding tests, using chemical additives as suggested in the literature, on a group of minerals including calcium carbonate, silica, nepheline syenite, talc and cement. Up to ten (10) different chemical additives were screened for each of these minerals. Details of the test methodology are described in this report.

4.0 REVIEW OF LITERATURE

4.1 General Discussion On The Mechanisms Of Chemical Additives

Chemical additives, commonly referred to as grinding aids, have found widespread acceptance in cement grinding other industrial minerals.

The primary benefits that chemical additives can impart during dry grinding are:

- Increases grinding rate or allows finer grinding
- Eliminates or retards the formation of aggregates, agglomerates, media and mill liner coating.
- Improves "dry flow" properties of ground material.
- Improves the dispersion of ground material.
- Improves air classification in closed circuit dry grinding applications.

Many minerals and cement when fine ground tend to re-aggregate or form agglomerates during grinding. Often this is accompanied by the formation of coatings on grinding media and mill walls and plugging of discharge grates.

Agglomeration causes poor flow through the mill, while media and liner coating due to the cushioning effect, results in poor grinding efficiency. In milling operations using closed circuits with air classifiers, classification efficiency is adversely affected by poor dispersion of the material to be classified. Agglomerates present in materials to be classified are rejected by the classifier as coarse particles and returned

to the mill. This results in the formation of more agglomerates and eventually halts the grinding process.

The formation of agglomerates and the particle size at which it exhibits itself varies from mineral to mineral. The type of grinding equipment also has a bearing on this phenomena. Mills with a high length to diameter ratio and high mill retention times, such as tube mills, tend to be more susceptible to agglomeration.

In general, (depending on the mineral) the finer the grind, the higher the probability of agglomerates and coatings to form. Overgrinding is therefore the primary cause for development of these problems.

Mechanical changes in the milling process can often reduce the severity of the problem. Increasing the open area of discharge grates can decrease the residence time. The weight ratio of grinding media to material can also be important.

Chemical additives can be employed successfully in many grinding operations where agglomeration and media or mill coating are observed. The selection of a chemical additive must be based on its effectiveness to eliminate agglomeration, mill and media coating and on the properties of the ground product. It is important to consider the effect additives may have on product performance. Laboratory testing of a number of different chemical additives is therefore essential.

Several procedures have been suggested in the literature and will be described in this report. The choice of chemical additives is relatively broad. They are commercially marketed as grinding aids. Others are simple chemical compounds readily available from chemical suppliers. Many potential chemical additives are discussed in the literature review, which is part of this report.

4.2 How (Why) Do Chemical Additives Work

There is some controversy as to the actual mechanics of how a chemical additive functions. There are two (2) basic theories that have been expounded by various researchers.

The first theory of how the chemical additive works is attributed to the so called Rehbinder effect, which stated in its simplest form suggests that:

- Improvements obtained in dry grinding in the presence of a grinding aid is caused by the adsorption of the surfactant on freshly produced surfaces. Thereby, reducing the surface free energy of the solid. It is further suggested that it enhances crack propagation.

Khodakov and Rehbinder [4] also suggested that during grinding an amorphous layer is formed on the surface of brittle materials. The chemical additive acts to reduce the thickness of this amorphous layer improving the fracture rate. Samasunduran[5] concluded, like Rehbinder, that a decrease in particle density occurs due to formation of a deep amorphous layer on the particles in prolonged grinding.

The second theory on how chemical additives function is based on their ability to reduce or eliminate static electrical charges of opposite polarity.

Pearson[6], Krupp, Pietsch and Rumpf 1967 and Rumpf 1962 all suggested that adhesion of particles of finely ground materials to each other, grinding media and mill liners is caused by Van der Waals Forces (Molecular attraction) and electrostatic charges on freshly produced surfaces.

Mardulier[7] noted the presence of static charges on freshly ground cement under conditions of low humidity. It was found in every instant that these charges had the same polarity, thus causing the particles to repel each other, but to stick to a dissimilar surface bearing the opposite charge. He concluded (in the case of cement) that static electrical charges developed by friction during grinding do not account for the formation of agglomerates during grinding.

Ocepek and Eberl[8] concluded from their studies that the Rehbinder effect plays a subordinate role. They attributed the positive effect of chemical additives on the chemisorption of the chemical on active surfaces and free valences of the particles resulting in reduced formation of agglomerates and media and mill wall coating.

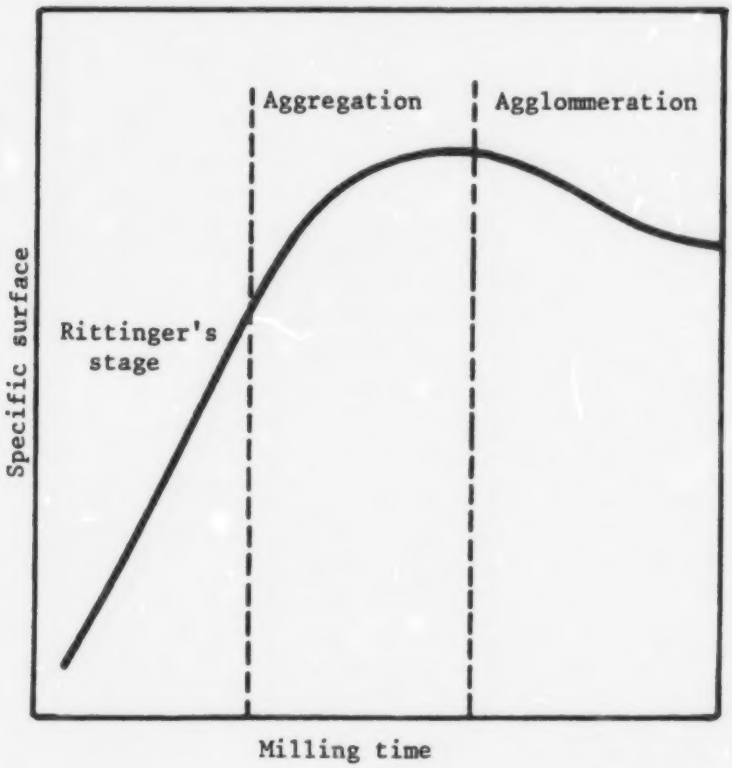
Smith[9] listed the significant forces acting on particle surfaces together with the size region where each force is operable. He concluded that Van der Waals, electrostatic and adsorption are experienced at the finer sieve sizes and below, while chemisorption was active at below 2 micron and valency forces at submicron sizes.

Rumpf[10] defined the particle size area and adhesion forces for three (3) specific areas. These being the particle size ranges which are affected by Van der Waals adhesion, chemisorb bonding, and valency bonding. He also concluded that valency and chemisorption forces were only in evidence at extremely fine sizes, but that Van der Waals forces are operable as coarse as 100 micron.

Opoczky[11] defined three (3) stages of grinding (Figure 1) as follows:

- The nominal grinding phase or Rittenger's stage which is followed by coating of the coarser particles by fines produced during grinding. In this (second) stage aggregates are formed due to Van der Waals charges which can be mechanically reversed since the energy involved amounts to 0.04 - 4 KJ/mole. On prolonged milling a third stage in which agglomerates are formed resulting from energy levels in the range of 40 to 400 KJ/mole. He considered the latter stage to be irreversible.

Figure 1



Opoczky further suggested that grinding aids are only effective during the aggregation phase, or Van der Waals phase.

Bhatia[12] studied the use of chemical additives in cement grinding. He explained the action of grinding aids as chemisorption of the additive on the reactivity centres of newly formed particles surfaces. This results in a reduction of the surface energy thereby eliminating the driving force that causes agglomeration.

Most of the scientific data points to the fact that grinding aids prevent formation of agglomerates and media coating, thereby improving the effectiveness of the grinding process and energy utilization.

There is evidence that chemical additives can be effective in grinding operations where no agglomeration or ball coating is present. Mardulier[13] states that an improvement in productivity of twelve (12) percent was noted in the grinding of a type I cement in a mill that normally experienced no problems with agglomerates or coating. This indicates that chemical additives can be effective in improving grinding rate. It is not unlikely that the grinding improvement was caused by an improvement in the dry dispersion of the ground material, which in turn improved air classification efficiency. (Authors comment)

Rose and Sullivan[14] reviewed most of the work undertaken in the field of grinding aid research prior to 1950. Snow[15] has summarized the implications of selected references (1973). Hartley, Pridbey and Wilk[16] prepared an updated synopsis of grinding aid literature in 1976.

Out of all these studies has come a bewildering array of hypotheses to explain the mechanism of grinding aids. Prevention of particle agglomeration and media coating as well as reduction of the strength of porous rocks by pre-soaking in liquids have been suggested. Klimpel and Manfroy[17] based on their research and review of work by others consider it highly improbable that the Rehbinder and Kalinkovskaya 1932 theory of chemomechanical effects could explain the mechanism of grinding aids in ball mills.

4.2.1 Observations

Despite the extensive amount of work conducted over the years there is still no precise, well stated theory, or factual evidence that clearly establishes the mode of operation of chemical additives used as grinding aids.

It is suggested by the author of this report that most research was conducted on a specific material under controlled conditions. Conclusions were drawn, or theories advanced on the basis of working with a single type of ore or a cement of a specific chemical composition. Different conclusions may have been made if the research work had been conducted with a multitude of different materials under the same controlled conditions. Other than the theoretical considerations of how a grinding aid functions, there are the practical considerations of the milling equipment employed: i.e. geometry of mills, residence time, ball-to-material ratio's, circulating loads and open and closed circuit operation.

Despite the lack of a satisfactory explanations as to how and why chemical additives perform as a grinding aid, there is ample practical evidence that many chemical additives can significantly improve the performance of a grinding operation, thereby significantly enhancing energy utilization.

As stated, chemical additives can be used to achieve different objectives. These are:

- Increased production of product at equal energy input.
- Equal production of product at finer particle size at equal energy input.
- Improved flowability, reduced pack set and conveying characteristics.
- Improvements in the efficiency of air classifiers.

4.3 Selection Of Chemical Additives

Most researchers are unanimous that polar liquids or compounds are the most effective chemical additives. Polar compounds are defined as having a union of atoms in which the bonds are electrostatic attractions between particles or ions with opposite charges. Anti-static agents should also be considered if there is evidence that static charges are present on the ground particles.

Eberl[18] tested tri-ethanolamine, ethyleneglycol, pinothane and HEA-2 a commercial grinding additive which is an ethanalomine acetate on several types of clinker. He concluded that these grinding aids were most effective on the harder type of clinker and that the efficiency increased for cements of higher surface area.

Ocepek and Eberl[8] studied HEA-2 in the grinding of calcite in a laboratory mill. They used a very narrow size fraction as feed material being 0.8 X 0.63 mm. After equal grinding time, the test with HEA-2 gave a significantly finer product with a 24.5% increase in the amount of material finer than .023 mm (23 micron).

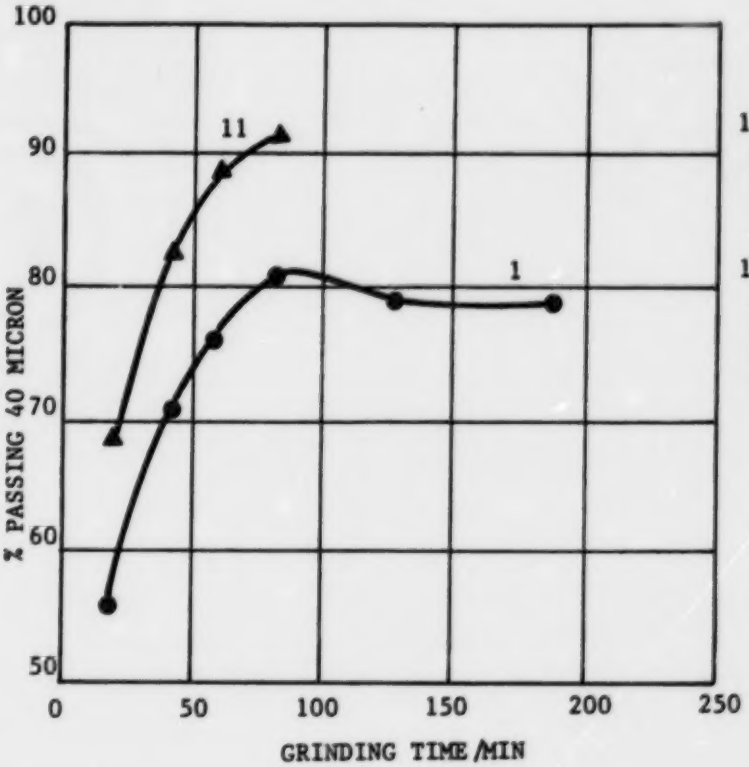
Von Dombrown, Hoffmann and Scheibe[19] conducted a detailed study with a large number of chemical additives. They particularly studied the effect of chain lengths of monocarboxylic acids in the grinding of limestone. Their results show a significant effect on grind as well as flowability. The shorter chain length: i.e. The number of carboxylic groups gave significantly better grinding rates and flowability than with chain lengths containing more than twelve carboxylic groups. For the particular material studied, monocarboxylic acid with a six (6) carboxylic groups i.e. C_6 gave optimum results with respect to fineness and flow properties.

They also related the effect grinding aids had on the performance of air classifiers and the effect grinding aids have on mass flow rates in a closed circuited grinding mill. Air classification was improved measureably.

Locher and Seebach[20] studied grinding aids introduced as vapour. Cement and other minerals were tested. They concluded that only if the vapour became chemisorbed on the particle surfaces did they function as a grinding aid by reducing the adhesive forces in the material and consequently prevented formation of coatings. Their study included the vapours of ethylene glycol, propylene glycol and butylene glycol. The correlation between the amounts adsorbed and the agglomerative forces between fine particles exposed to the adsorbents was very pronounced.

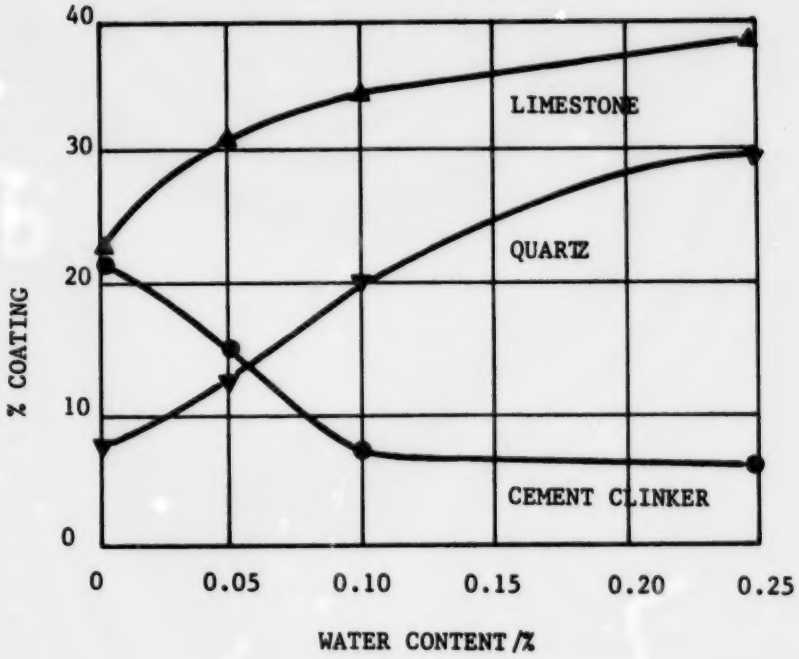
Scheibe, Dalmann and Rosenbaum[21] conducted a very detailed study on a large group of chemical additives for their functionality as grinding aid in the grinding of cement, quartz and limestone. They specifically concentrated their attention on the influence of mill wall and media coating on the fineness of grind produced. It was found that if limestone was ground without an additive, mill and ball coatings were forming and after 75 minutes the maximum fineness was achieved (80% - 40 micron), further grinding for up to 200 minutes did not result in a finer product, giving conclusive proof that mill wall and media coating caused stoppage of the grinding process. In comparison, the same material was ground in the same mill but the mill was stopped every two (2) minutes and the media and mill wall cleaned with a brush: i.e. mechanical removal of the coating. Results showed a significantly higher grinding rate. After 75 minutes a fineness of 91% passing 40 micron and the grinding curve was not flattening out (Figure 2).

Figure 2



They also studied the effect of water addition on mill and media coating during the laboratory grinding of limestone, quartz and cement. Tests were conducted at 0.0% - 0.5%, 0.10% and 0.25% water addition for each of these materials and one (1) hour grinds. It was found that in the case of quartz and limestone there was a significant increase in coating with increasing water content, while with cement, the degree of coating decreased significantly as compared to no water addition. A minimum level of coating was achieved at 0.10% water addition. Further addition had no effect on coating. It was assumed that hydration of the cement was the reason for a reduction of mill and media coating (Figure 3).

FIGURE 3



These authors studied some twenty five (25) chemicals with representative groups of anionic, cationic and non-ionic chemicals. They studied the amount of minus 40 micron produced and the Blaine surface area as a function of additive and rate of addition. Their results show conclusively that in the case of cement, the additive that produced the highest amounts of minus 40 micron did not have the greatest surface area or vice versa. The study also established optimum addition levels for each additive. Similar studies conducted with quartz and limestone found a number of effective additives for each of these minerals.

Deckers and Stettner[22] studied the effect of mill conditions on the functionality of grinding aids. They concluded that grinding aids remain effective during changes in operating conditions of the mill. They also found that the magnitude of the effect and quantity by which an optimum effect is achieved may differ.

They concluded that changing the type of grinding media or charge volume had a pronounced effect. They expressed their results in terms of improvement in energy utilization by measuring the surface area produced per kWh. They compared three (3) types of grinding media, conventional steel balls, cylindrical (Cylpebs) steel media and fused alumina balls, with various grinding aids. Results on quartz milling show conclusively that improved energy utilization is a function of the type of grinding media and the grinding aid as shown in the following table.

Table 4-1

PERCENT IMPROVEMENT IN ENERGY CONSUMPTION*
COMPARED TO GRINDING WITHOUT ADDITIVES

	Type Of Grinding Media		
Grinding Aid	Steel Balls	Steel Cylinders	Aloxide Balls
Fatty Acid	40.6%	75.9%	14.3%
Mono-Ethylene Glycol	15.2%	19.0%	15.1%
Dolapix P.C.N.	13.7%	16.6%	5.9%
Distilled Water	8.6%	22.4%	9.2%
Diesel Oil	7.9%	18.9%	26.5%
Tri-Ethanolamine	4.9%	17.3%	31.6%

* From Deckers, M., and Stettner, W. - Aufbereitungs-Technik No. 10 - 1979

These results show conclusively that grinding media and the grinding aid have a decided effect on the energy utilization. Results with fatty acid show significantly greater energy utlization with steel cylinders compared to the other media, while with tri-ethanolamine the best improvement was obtained with aluminum oxide.

This further demonstrates that energy utilization is a function of combining proper grinding media selection with the right additive. Optimum selection may not always be possible depending on product requirements, and milling equipment.

Schneider[23] conducted detailed studies on the grinding of cement at 100°C to 120°C using various grinding aids. He compared the Blaine surface area in cm^2/gram obtained at various milling times for various additives. Tests were conducted in a batch mill.

Two hours of grinding cement clinker without a chemical additive resulted in a Blaine surface area of 3700 cm^2/gram. Amine acetate and ethylene glycol as additives gave Blaine surface areas of 4250 and 4300 cm^2/gram respectively after two hours of grinding. Propylene glycol gave the best result with a Blaine surface of 4550 cm^2/gram after two hours of grinding. This translates to an energy consumption of 78 kWh/tonne for no grinding aid, 48 kWh/tonne for grinding with propylene glycol at an equal Blaine surface area of 4500cm^2/gram and 56 kWh/tonne and 34 kWh/tonne at a Blaine surface area of 3750 cm^2/gram for no grinding aid an propylene glycol respectively.

Further tests with ethylene glycol in a 1.2 meter X 4.2 meter cement mill at different levels of addition gave the following results:

Table 4-2

CEMENT GRINDING TESTS WITH ETHYLENE GLYCOL*

MILL FEED, BLAINE IS 2800 cm^2/GRAM

Ethylene Glycol Addition %	Surface Area cm^2/g	Production T.P.H.	Increase In %
-	3770	2.0	-
0.009	3880	2.2	10
0.02	3850	2.4	20
0.03	3840	2.7	35
0.05	3770	3.0	50
0.1	3780	3.9	95
0.1	4430	2.0	100
-	4440	1.0	-

These results clearly show how, under closely controlled conditions, increases in productivity of up to 100% were achieved in this mill using a preground cement clinker with a Blaine surface area of 2800 cm^2/gram at various addition rates of ethylene glycol.

Schneider[23] also gives detailed results for a large number of cement mills using additives. These are all mills in actual cement production, and are of the circulation or open circuit type. Following is a summary of this data for circulating mills. Results are expressed as the surface area produced per kWh.

* After Schneider , H. the use of grinding aids in cement grinding Zement-Kalk-Gips 1969 #5 pp 193-201

Laapas, Lahtinen and Lukkarian[24] studied the use of grinding aids in the dry and wet fine grinding of talc, quartz, calcite and wollastonite. They selected sodium oleate for quartz, tri-ethanolamine for calcite a salt of polycarboxylate for talc

Table 4-3

ACTUAL EXAMPLES OF INCREASED PRODUCTIVITY WITH CLOSED CIRCUIT CEMENT MILLS

Mill Size M X M	Surface Area cm^2/gram	Additive	Addition Rate	% Productivity Increase	Surface Area Produced In 10^6 cm^2/kWh Without Grinding Aid	With
3.0 X 10.5	4850	Tri-ethanolamine	0.03	15	69	78
3.6 X 11.5	4900	Ethylene Glycol	0.05	22	72	88
2.4 X 14.0	5000	Ethylene Glycol	0.05	43	54	77
2.4 X 14.0	5000	Propylene Glycol	0.05	55	54	83
3.4 X 10.6	5100	Ethylene Glycol	0.08	28	55	70
3.4 X 10.6	5100	Propylene Glycol	0.08	45	55	81
2.4 X 14.0	5300	Ethylene Glycol	0.09	15	64	73
2.6 X 15.0	5500	Propylene Glycol	0.09	28	43	54

After Schneider, H. - The use of grinding aids in cement grinding - Zement-Kalk-gips (Cement, Lime and Gypsum) 1969 #5 pp 193-201

and sodium hexametaphosphate for wollastonite. They reported that these additives performed well in both wet and dry grinding with the exception of sodium hexametaphosphate which was only functional in wet grinding of wollastonite.

They tested these additives in a conventional laboratory tumbling, vibratory and attrition mills. They concluded that in a tumbling mill, the grinding aids showed the best performance in fine grinding at sizes below 15 microns but were not effective at coarser grinds. Their results were presented as the amount of energy consumed per unit of surface area and the median particle diameter (D50) of the product relative to energy consumption. In dry grinding significantly higher surface areas were observed for minerals ground with the additives except wollastonite. Energy consumption was reduced per unit of surface area or the amount of energy required to achieve a given median particle size (D50).

The authors concluded that at coarser grinds, high fluidity of the ground material in fact reduced the grinding rate in a tumbling mill. The increased fluidity of the material likely reduced mill residence time. i.e. it contributed to an increase in the circulating load. A reduction in the open area of the discharge grate would likely correct this (authors' comment).

The authors also found that dry grinding in a highly aggregated state, resulted in increased surface area without formation of new fine particles. This was attributed to formation of micro-cracks in the particles. Surface area measurements were all made by gas adsorption. This method of surface area determination is more likely to show this phenomena than the typical Blaine surface area determination.

Scheibe, Hoffman and Dombrowe [25] investigated the effect of grinding aids on the fineness and other properties of cement. Examinations were made in a laboratory mill and in an industrial tube mill of 2.4 X 13 meters. Tri-ethanolamine, or residues of glycol synthesis were used at a rate of 0.01 to 0.05% added as drops or spray. The authors concluded that industrial conditions can be modeled with a laboratory mill. They also concluded that additives increased fineness and decreased coating on mill walls and grinding media. Method of additive addition had little influence on the results. It was also found that dust collected by the dust collector increased in volume and the particles size was finer. To minimize the influence of temperature, tests conducted with the laboratory mill were done at 180°C, which is significantly above the 80-150°C temperatures typically encountered in cement grinding. The primary objective was to establish grinding additives which are functional at these temperatures.

Pit and Quarry[26] reviewed the history of the use of cement grinding aids and gives examples of results obtained with UCAR-CGA, a commercial grinding aid - pack set inhibitor. The work conducted in the laboratories of Union Carbide showed a significant increase in Blaine surface area and reduction in pack set index as shown in the following table.

Table 4.4

CLINKER AND 3.5% GYPSUM

Additive %/Wt	Blaine Surface cm^2/gram	Pack Set Index
0.0	3500	13
0.02	3770	5
0.033	3870	2
0.05	3900	1
0.10	4080	0

The pack set index is a measure of the flowability of cement. This is determined by vibrating 80-100 grams of cement under specified conditions in a 250ml flask. The flask is then turned on its side and rotated around its axis until the compacted cement collapses. The number of 180^{o} turns is an expression of the energy required to collapse the cement and is assigned the numerical value of the pack set index. The lower the number the lower the pack set. Cement with pack index values in the range of 15-25 are considered to have potential flow problems while above 25 the cement is usually unsatisfactory.

Tests were also conducted on the effect of the grinding aid on the compressive strength of the cement according to A.S.T.M. Test C-109 on type I, II and III cement used in mortar, or test A.S.T.M. C-39 for type I, II, and III cement used in concrete. In all cases, increased compressive strengths for 1, 3, 7 and 28 day curing tests in mortars and 3, 7, 28 and 90 day curing in concrete were observed.

British patent 1413395[27], filed in 1972 by Pluess-Stauffer, AG of Switzerland involves the use of an aliphatic ester of acetic acid in the grinding of chalk or calcite. Claims, as made in the patent, show that in a ball mill of $19.M^{3}$ volume, containing 22 metric tons of 16mm steel balls, mill output at the required particle size distribution was from 6 to 7 tonnes per hour. An addition of 700 ppm (parts per million) of the chemical additive increased productivity to 10 tonnes per hour at equal particle size.

A second example in the patent describes the improvement in the operation of an air classifier when classifying material has been ground with the additive. It showed that when the classifier was fed with four (4) tonnes per hour of untreated material the yield of specification product was two (2) tonnes per hour.

When the same material was treated with 250 ppm of the additive at the same feed rate of 4.0 tonnes per hour, a productivity rate increased to 2.4 tonnes per hour, an increase of twenty (20) percent.

All of the foregoing, shows that there is a significant amount of scientific evidence that chemical additives used in dry grinding of cement or other minerals can be effective in improving energy conservation.

Increased productivity at equal particle size results in a significant reduction of energy consumption expressed in kWh/ton of product. Alternatively, the use of chemical additives allows finer grinding at equal output and energy consumption.

The literature, as reviewed, gives a strong indication that milling conditions such as mill speed, media charge, residence time, etc., are variables that could affect the performance or effectiveness of a chemical additive.

Evidence is conclusive that benefits derived from the use of chemical additive are a function of the degree of fineness required and the particle size at which any given material will start to form aggregates, agglomerates or cause mill and media coating. This may exhibit itself at different particle size distributions for any given mineral or cement or for chemically and mineralogically identical materials from different deposits or the same material ground in a different type of mill.

Grinding systems operating in a closed circuit with air classification equipment will show an additional benefit in the more efficient operation of the air classifier if the material to be classified is well dispersed and free of aggregates or agglomerates.

The necessity of testing potential chemical additives under laboratory conditions is self evident. Initial selection of a chemical additive must also be based on what potential side effects a certain additive may have on product performance.

This is true for cement as well as minerals to be used as fillers/extenders in paints, plastics or in ceramic applications.

Practical use of chemical additives in grinding may also necessitate modifications to the grinding equipment or mode of operation. Increased flowability of the material to be ground can lead to a significant reduction in mill residence time. This, in turn, can cause an increase in the circulating load and in the case of closed circuit grinding in overloading the air classifier with a corresponding loss of classification efficiency. Adjustments to discharge openings or changes in media volume can reduce these effects.

In summarizing, the potential benefits that can be realized from the use of chemical additives are most likely to be realized in grinding operations that suffer from overgrinding, agglomeration and the formation of coatings on mill walls and grinding media.

Such conditions lead to a further reduction in energy utilization of an already inefficient process. Use of properly selected chemical additives can restore grinding efficiency to the level predicted from theoretical evalutation and from the Bond work Index of the material.

In some instances it has been observed that in grinding operations not experiencing any difficulties with the disruptive effects of agglomeration or coatings an improvement in grinding is realized when chemical additives are employed. This gives some credence to the theory that chemical additives may accelerate the grinding process, by means other than preventing aggregation and formation of coatings.

5.0 INDUSTRY QUESTIONNAIRE

As part of this project on the use of chemical additives, a study was undertaken to establish the extent of practical application of chemical additives in grinding cement and other industrial minerals.

With the assistance of officials from Energy, Mines and Resources (EMR), a questionnaire was designed for distribution to a randomly selected number of cement and other industrial mineral producers in North America, Europe, Mexico, South Africa and Japan.

The recipients were asked to complete the questionnaire and return it to Energy Mines and Resources, in Ottawa. The purpose of this was to remove any company identification in order to protect the confidentiality of the responding company.

A total of one hundred forty six (146) questionnaires were sent out. A total of forty six valid responses were received (a 31.5% response rate).

All of the responses were evaluated and the information provided was classifed into several categories.

The following questions were asked in the questionnaire.

QUESTIONNAIRE

1. Is your company now or has it in the past used chemical additives in dry grinding to enhance the efficiency of grinding or air classification? Yes No
   If no, proceed to Item #13, Page 3.

   If so, are you using these chemical additives to improve one or more of the following?

   a. Increase grinding rate ( )
   b. As flow aid ( )
   c. As dispersant ( )
   d. Improved air classification ( )
   e. Other_____

2. What mineral(s) do you grind and to what size?

   Minerals: a. _____

   b. _____

   c. _____

		Minerals		
		a.	b.	c.
100% passing size:	74 micron	( )	( )	( )
	45 micron	( )	( )	( )
	30 micron	( )	( )	( )
	20 micron	( )	( )	( )
	10 micron	( )	( )	( )
	finer	( )	( )	( )

3. Do you operate grinding mills in closed circuit with classifiers? Yes No

4. What type of classifiers do you employ?

   a. Standard type air classifiers ( )
   b. High efficiency classifieres ( )

5. What was your primary consideration for using chemical additives?

a. Energy conservation ( )
b. System productivity - efficiency ( )
c. Product quality ( )
d. Improved air classification ( )
e. Eliminate static charges ( )
f. Prevent agglomeration ( )
g. Improved product performance ( )
h. Other:_____

6. Has the use of chemical additives in dry grinding provided you with any of the following?

a. Less kWh per ton of product ground ( )
b. Ability to produce finer products ( )
c. Better control over grinding process ( )
d. Improved air classification efficiency ( )
e. Other:_____

7. Has the use of chemical additives resulted in lower production costs i.e. $/ton? Yes No

% Saving ( )

8. Do you use chemical additives that your company has developed? Yes No

9. Do you purchase your chemical additives? Yes No

10. What type of chemical additives do you use? (generic name)

_____

11. What is the typical addition rate of additive per ton of fresh feed?

_____

12. We have used chemical additives in dry grinding and concluded the following:

a. They did not enhance grinding ( )
b. They affected product performance ( )
c. Did not result in energy or other savings ( )

NON USERS OF CHEMICAL ADDITIVES IN DRY GRINDING

13. Are you familiar with the use and potential of chemical additives to improve dry grinding and or air classification? Yes No

14. Have you considered the use of chemical additives in dry grinding? Yes No

15. What would be your primary concern regarding the use of chemical additives assuming they provide you with better productivity, i.e. tph and lower energy cost/ton?

    a. Cost of chemical additives ( )
    b. Effect on product performance ( )
    c. Method of application ( )
    d. Other:_____

16. In order to consider the use of chemical additives I would require knowledge of the following:

    a. What are the potential benefits ( )
    b. How do I select and additive ( )
    c. How do I measure performance ( )
    d. What is the effect on my end products ( )
    e. Other:_____

5.1 Response To Questionnaire

Total number of questionnaires sent out	146
Number of questionnaires sent to cement manufacturers	101
Number of questionnaires sent to other industrial mineral producers	45
Valid response from cement companies	35
Valid response from non-cement companies	11
Percent response from cement companies	35
Percent response from non-cement companies	24
Responding cement companies not using or not familiar with chemical additives	0
Responding non-cement companies not using or not familiar with grinding aids	10

VALID RESPONSES TO QUESTIONNAIRE
BY COUNTRY

CEMENT

United States	18
Canada	11
Japan	1
Not Identified	3
Mexico	2
	35

MINERALS

Canada	7
United States	1
Italy	1
Norway	1
	10

5.2 Analysis of Response to Questionnaires

In several instances not all of the questions were answered.

It is significant to note that all of the responding cement companies (35) indicated that they are, or have used chemical additives, while all of the respondants from the industrial minerals industry (11) are not, or have not used chemical additives.

It is the authors opinion that many of the industrial minerals companies that did not respond to the questionnaire are in effect using chemical additives, but, may not have wished to disclose this, despite the confidentiality.

The number of responses from the cement industry was sufficient to provide a good cross section of the extent and reasons for using chemical additives.

CEMENT INDUSTRY QUESTIONNAIRE

1. Is your company now or has it in the past used chemical additives in dry grinding to enhance the efficiency of grinding or air classification?

   Yes 34 No 0

   If so, are you using these chemical additives to improve one or more of the following:

a.	Increase grinding rate	28
b.	As flow aid	31
c.	As dispersant	8
d.	Improved air classification	6
e.	Other: dissipate static on grinding balls	1
	increase setting time	1
	reduce lumping in silos	1

2. What mineral(s) do you grind and to what size?

Minerals:	a. Cement	33
	b. Gypsum	10
	c. Slag, Limestone, Shale	4

		Minerals		
		a.	b.	c.
100% passing size:	74 micron	27	8	
	45 micron	5	2	
	30 micron	0	0	
	20 micron	1	0	
	10 micron	0	0	
	finer	0	0	

3. Do you operate grinding mills in closed circuit with classifiers?

   Yes 34 No 0

4. What type of classifier do you employ?

a.	Standard type air classifiers	32
b.	High efficiency classifiers	6

5. What was your primary consideration for using chemical additives?

a.	Energy conservation	11
b.	System productivity - efficiency	22
c.	Product quality	11
d.	Improved air classification	3
e.	Eliminate static charges	13
f.	Prevent agglomeration	10
g.	Improved product performance	5
h.	Other: Aids dry flowability	1
	Eliminate pack-set	2

6. Has the use of chemical additives in dry grinding provided you with any of the following?

a.	Less kWh per ton of product ground	27
b.	Ability to produce finer products	10
c.	Better control over grinding process	14
d.	Improved air classification efficiency	7
e.	Other: Elimination of pack set	4
	Aids dry flowability	5
	Improved setting time	1
	Reduces lumping in silos	1

7. Has the use of chemical additives resulted in lower production costs i.e. $/ton?

Yes 25 No 7

% Savings

1%	1	3%	2	7%	1	15%	1
2%	3	5%	2	10%	3	20%	1

8. Do you use chemical additives that your company has developed?

Yes 4 No 30

9. Do you purchase your chemical additives?

Yes 33 No 1

10. What type of chemical additives do you use (generic name)?

Glycol	17	Graid I	2
Amines	13	Busperse 49C	1
Phenols	2	MHEA-2	5
Acetic Acid	2	GA 100	1
Aromatic Hydroxides	2	Grace DDA7	1
VCAR	1	REAX G-2	1
CGA%	1		

11. What is the typical addition rate of additive per ton of fresh feed?

0.005 - 0.014%	4	0.050%	1
0.015 - 0.020%	5	0.100%	1
0.021 - 0.030%	10	0.260%	1
0.040%			

12. We have used chemical additives in dry grinding and concluded the following:

a. They did not enhance grinding	4
b. They affected product performance	13
c. Did not result in energy or other saving	4

6.0 LABORATORY TESTS OF CHEMICAL ADDITIVES

WHAT FACTORS MUST BE CONSIDERED IN SELECTING A CHEMICAL ADDITIVE IN DRY GRINDING.

1. Each chemical additive must be thoroughly tested in bench scale experiments and in a dynamic production environment.

2. Grinding aids recommended for use with a certain mineral may perform better with material from one deposit than another. Test several types.

3. What effect (positive or negative) can or may chemical additives have on products produced, relative to their intended industrial application.

4. Cost of additive, relative to the incremental increase in productivity or reduced energy cost. Will there be a saving?

WHICH MINERALS ARE LIKELY TO BENEFIT MOST FROM CHEMICAL ADDITIVES - BASED ON MINERALS TESTED

1. Cement

2. Carbonate Minerals - limestone, calcite, dolomite

3. Silica - Quartz, Quartzite, sandstone

4. Feldspathics i.e. feldspar, nepheline syenite

IN WHAT GRINDING SYSTEMS CAN CHEMICAL ADDITIVES BE MOST EFFECTIVE

1. In long tube mills with gravity discharge or conventional ball or pebble mills with relatively long retention times.

2. In systems where very fine grinding is required to meet product specifications and where the oversize returned to the mill is quite fine relative to the size of fresh mill feed.

3. In grinding systems with an air classifier operating in closed circuit with the milling equipment.

4. Grinding minerals with poor flow characteristics, high pack set index or minerals that tend to agglomerate at relatively coarse particle size.

IN WHAT MILLING SYSTEMS ARE CHEMICAL ADDITIVES NOT LIKELY TO BE EFFECTIVE

1. Fluid energy mills

2. Dry grinding rod mills

3. Hammermill or cage mills

4. Stud mills

No published data was obtained on the use of grinding aids in roller mills. Roller mills may benefit from additives that act like flow aids, primarily to improve the dispersibility of the ground product prior to air classification.

To test the concept of chemical additives in dry grinding of non-metallic industrial minerals, a series of laboratory experiments were conducted with a wide range of potentially suitable additives.

For these tests the following minerals were selected:

- Limestone (calcite)
- Silica - Quartzite
- Nepheline Syenite
- Talc
- Cement - Clinker

The purpose of the bench scale test was to observe the grinding rate, occurrence of media and mill coating and formation of aggregates or agglomerates.

The primary objective was to establish which chemical additives gave the best overall results with respect to the degree of fineness achieved in the specified time period. In each case, tests were comparative with the same material ground without additives.

Bench Scale Testing

6.1 Methodology

6.1.1 Equipment

- One (1) gallon ceramic jar mill
- Mill drive - variable speed roll drive
- Sieves with stainless steel cloth of 325 mesh (45 micron) openings
- Tyler roll tap sieve shaker

6.1.2 Grinding Media

- o 3/4" fused alumina balls
- o Weight 4,000 grams

6.1.3 Sample Size

900 gram - 20 X 100
- 40 X 100

6.1.4 Mill Speed

74% of critical

6.1.5 Grinding Period

Total 3.5 hours broken down in 7 - 30 minute cycles

6.1.6 Sampling

50 gram of ground material removed after each cycle for size analysis i.e. % retained on 325 mesh (45 micron)

Sample replaced with 50 grams of product from a duplicate test.

6.1.7 Method of Grinding Aid Addition

Method of addition was by weight. The appropriate amount of grinding aid was premixed with 100 grams of mill feed which was then added to the mill charge.

6.2 Description of Test

For each mineral tested a duplicate grinding test was conducted. The mill drive was equipped with a timing device set for thirty (30) minutes.

At the end of each thirty minute cycle the mill was opened and the charge removed by screening over a 1/2" screen. A fifty (50) gram sample was removed for size analysis.

This sample was replaced with fifty (50) grams of material taken from the duplicate test. In this manner the weight of the sample in the mill was kept constant at 900 grams. To the duplicate test mill, 50 grams of fresh feed was added, and the second grinding cycle started. This was repeated for a total of seven (7) cycles with a total duration of 3.5 hours, with the exception of the tests on cement which was ground for four (4) cycles of thirty (30) minutes or two (2) hours.

6.3 Size Analysis

The fifty (50) gram sample removed from the test mill was placed on a standard 8" Tyler sieve with stainless steel cloth with a 325 mesh opening, equivalent to 45 micron. The sample was then washed with water to remove material finer than the sieve opening. The residue retained on 325 mesh was washed with acetone and then dried in an oven at 80°C in the sieve.

The sieve, containing the dried sample was then placed on a Tyler roll-tap sieve shaker for ten (10) minutes to remove any material finer than 325 mesh not removed during washing.

The residue of material coarser than 325 mesh (45 micron) was weighed and the percentage of material retained on 325 mesh (45 micron) calculated.

6.4 Method of Additive Addition

A predetermined amount of the additive was premixed with 100 grams of the material to be tested, using a blender this mixture was then added to the mill together with the remaining mill charges. This was done for the actual test mill and the duplicate mill.

A further 500 grams of mill feed was premixed with the appropriate amount of the additive to be usei as make-up feed for the duplicate mill.

6.5 Presentation of Results

Results from size analysis are summarized in tabular form for each of the minerals and chemical additives tested.

A second method of result presentation is given by plotting the percentage retained on 325 mesh (45 micron) versus grinding time on a graph.

For comparison purposes, each of the graphs shows the plotted results for material ground, with and without additives. By presenting results in graphical form, a clearer picture of the effect various chemical additives have on grinding is obtained.

6.6 Alternative Method

The method, as employed in the bench scale tests, is only one of several that can be used. In the test program, the primary objective was to observe how effective additives are in preventing mill and media coating, agglomeration and the ability to maintain or accelerate particle size reduction.

An alternative method is to grind for a specific time period. Remove the product from the mill and screen the ground product at a predetermined size, for instance 200 mesh (74 micron) comparing the weight of the material passing 200 mesh (74 micron) from the test with additive with the weight from the test without additive. Fresh feed equal to the amount of product removed, together with the appropriate amount of additive is added back to the mill. This cycle is repeated four to five times. The combined weight of product passing 200 mesh (74 micron) divided by the total grinding time is the number of grams of product produced per minute of grinding time. Effective grinding aids should show a higher rate. This method is effective for materials that exhibit agglomeration and/or ball and lining coatings at relatively coarse particle sizes.

An addition to this procedure is to grind for a time period that will result in an undersize percentage of 30 - 35 percent for each grinding cycle. The grinding time for each cycle is calculated on the basis of the amounts of undersize produced in the previous cycle. This is repeated for a total of five cycles. The combined weight of product divided by the total grinding time gives the number of grams of product per unit time. If the chemical additive is effective it should result in producing equal amounts of material in less grinding time.

A combination of these methods should provide a clear indication of the effectiveness of chemical additives and allows proper selection to be made. In addition to grinding, the effect of additives on the efficiency of subsequent air classification may also be determined.

7.0 GRINDING OF LIMESTONE WITH ADDITIVES

A total of eight (8) chemical additives were screened for their effectiveness as a grinding aid using the procedure as outlined in Section 6.2. The feed material was a 10 X 100 mesh soft limestone.

7.1 Test Results

Results from these tests are shown in table form in Table 7.1. Graphs comparing the obtained results obtained with each additives against the material ground without additives are given in Figures 7.1.1 to 7.1.9.

7.2 Interpretation of Results

7.2.1 Control Sample

The control sample, ground without additive showed formation of agglomeration and coating of mill walls after one (1) hour of grinding. Media coating started after 1 1/2 hours. Grinding rate was reduced significantly after this time. For optimum grinding results this particular material should not be ground finer than approximately eighty five (85) percent passing 325 mesh. Figure 7.1.1 shows the grinding rate curve for the control sample.

Table 7.1

BENCH SCALE - STATIC GRINDING AID TEST - PROJECT 5174-3

MATERIAL - 16 X 100 MESH LIMESTONE

Grinding Aid	Addition Rate %/Wt	Percent Retained on 325 Mesh (45 micron) at Time 0.5 Hr	1.0 Hr	1.5 Hr	2.0 Hr	2.5 Hr	3.0 Hr	3.5 Hr
No Grinding Aid	-	32.9	16.9	11.2	8.6	7.7	6.2	4.9
Tri-ethanolamine	0.5%	23.4	4.94	1.44	1.32	1.36	1.22	1.04
20% Acetic Acid	0.02%	23.2	6.42	2.36	2.48	1.70	2.00	2.00
Ethylene Glycol	0.02%	26.7	9.62	5.00	4.56	3.66	2.92	2.48
Alkaryl Sulphate 20% Solution	0.02%	27.7	12.7	8.32	1.66	1.55	1.43	2.24
60/40 Ethyl Alcohol	0.05%	13.7	8.76	3.11	2.02	1.82	2.24	2.00
Silicon Glycol Surfactant	0.02%	28.5	11.8	6.71	5.32	3.98	3.12	2.84
Acetic Acid - Tri-ethanolamine	0.02%	25.6	8.61	4.62	3.96	3.26	3.14	2.70
Alkasperse A-5	0.02%	28.1	11.0	6.28	3.60	3.92	3.60	2.78

7.2.2 Tri-Ethanolamine 0.05% By Weight

Figure 7.1.2 shows a very strong initial grinding rate. After one (1) hour the percent retained is equivalent to a three (3) hour grind without additive. After 1 1/2 hours, maximum fineness was reached and from this point on, no further size reduction was obtained. Mill walls and grinding media remained uncoated up to two (2) hours of grinding, but, agglomeration and coating developed after this time. The desired fineness of ninety-five (95) percent passing 325 mesh (45 micron) was achieved in one (1) hour. No agglomerates were noted in the ground product, which exhibited good fluidity.

7.2.3 Acetic Acid 20% - 0.1% By Weight

Figure 7.1.3 shows that acetic acid is effective in increasing or maintaining a high reduction rate. Up to ninety five (95) percent passing 325 mesh (45 micron), was achieved in little over one (1) hour. Grinding stopped completely after 1.5 hours. Formation of agglomerates was noted after 1.5 hours.

7.2.4 Ethylene Glycol - 0.02% By Weight

Figure 7.1.4 shows that ethylene glycol is effective up to ninety (90) percent passing 325 mesh (45 micron). Grinding stopped completely after 1.5 hours at which time coatings were noticed on mill walls and the grinding media. Agglomeration was noted after two (2) hours. Ethylene glycol is not as effective as the preceeding additives.

7.2.5 Alkaryl Sulphate - 0.02% By Weight

This additive showed a lower grinding rate during the first two (2) hours, after which no further grinding took place. The additive was effective in eliminating mill and ball coating. No evidence of either was noted until after 3.0 hours. The material had excellent fluidity. Results are shown on Figure 7.1.5.

7.2.6 60/40 Ethyl Alcohol - Acetic Acid Mixture - 0.05% By Weight

Figure 7.1.6 shows a very high initial grinding rate achieving better than ninety (90) percent passing 325 mesh (45 micron) in one (1) hour of grinding. Grinding stopped after 1.5 hours. Mill walls started to coat after 1.5 hours and grinding media after 2.0 hours. This was followed by the formation of agglomerates. This additive is effective for grinds up to 95% passing 325 mesh (45 micron).

7.2.7 Silicone Glycol Surfactant - 0.02% By Weight

Figure 7.1.7 shows the results from using a silicone glycol. This additive is an excellent dispersant, as shown by the fact that the mill walls and grinding media remained clean up to 3.0 hours of grinding with no evidence of agglomeration. While not as effective as a grinding aid, this additive may well be of value as a dispersant for improving air classification.

7.2.8 50/50 Mixture of Acetic Acid - Triethanolamine - 0.02% By Weight

This additive is effective in the early stages of grinding. Grinding stopped after 1.5 hours. This additive is effective in preventing mill coating for up to 2.5 hours and ball coating up to 3.0 hours. Material had good fluidity and showed no evidence of agglomeration. See Figure 7.1.8.

7.2.9 Alkasperse A-5 - 0.02% By Weight

Alkasperse A-5 is a polyacrylate. This type of chemical is effective as a grinding additive in the fine wet grinding of calcite at high slurry density. In dry grinding it may be effective as a dispersant - flow aid. This was not confirmed by the test. Re-agglomeration occurred after 2.0 hours of grinding. The grinding rate was only marginally better than the control sample. See Figure 7.1.9.

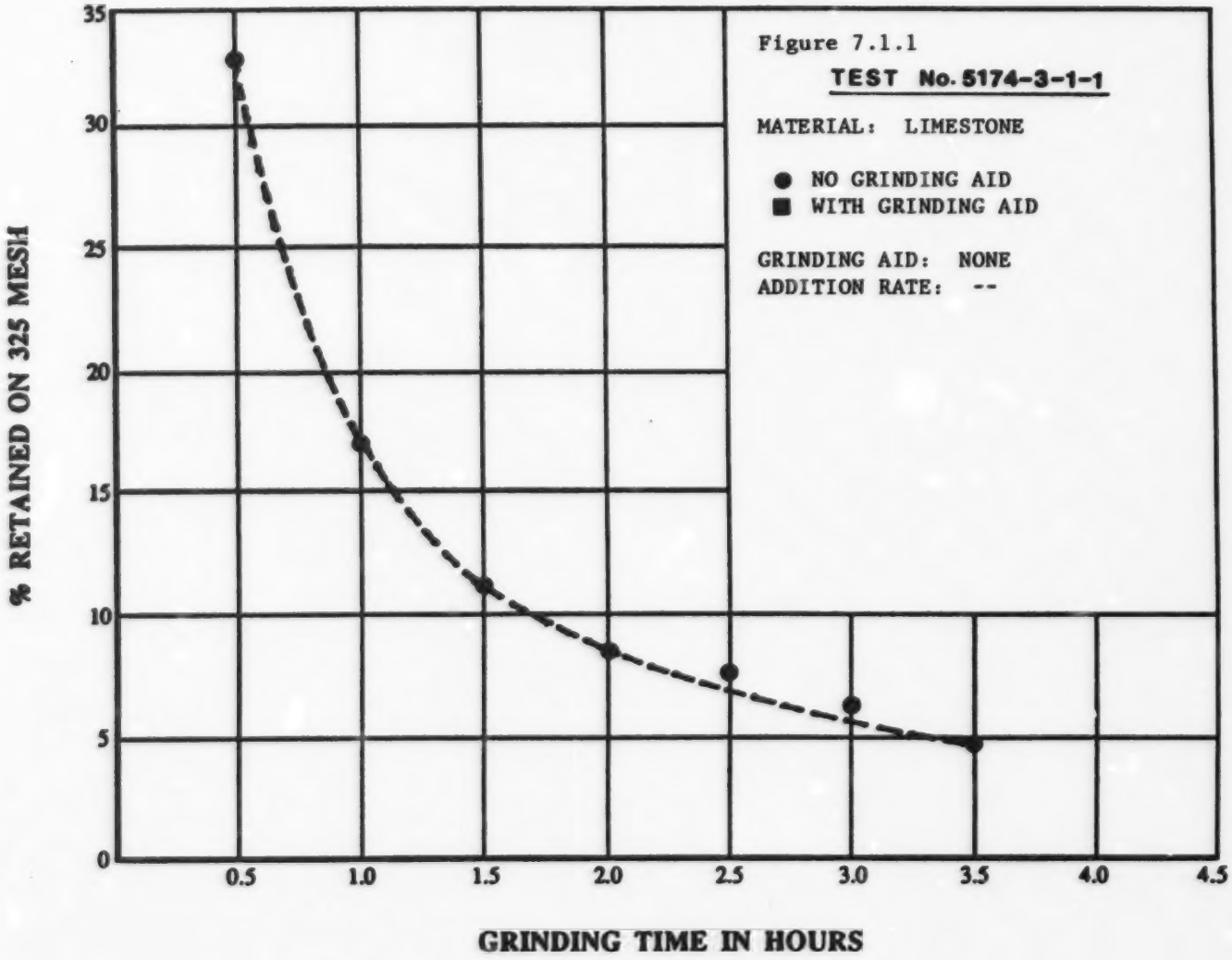
7.3 Conclusions on Grinding of Limestone

Several of the additives, specifically Tri-ethanolamine, a mixture of ethyl alcohol and acetic acid and a 20% solution of acetic acid were found to be effective as grinding aids with this particular type of limestone. Each of these three additives showed a high initial grinding rate, and achieved relatively fine particle size distribution before the onset of ball or mill wall coatings.

It has been the authors experience that results obtained with a particular type of limestone or calcite cannot be extrapolated to other limestones. Different results may well be experienced with other limestone.

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3



DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

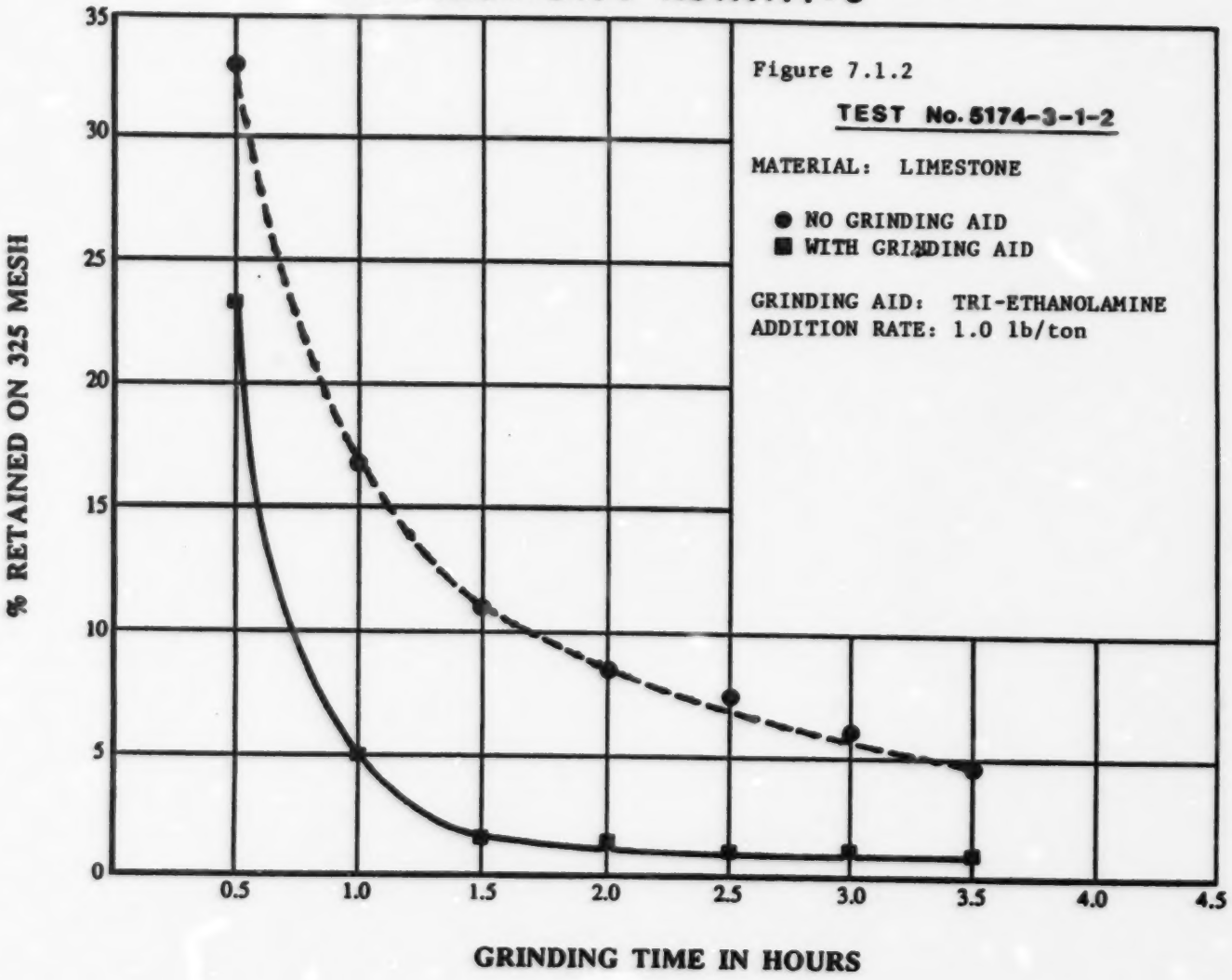


Figure 7.1.2

TEST No. 5174-3-1-2

MATERIAL: LIMESTONE

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: TRI-ETHANOLAMINE
ADDITION RATE: 1.0 lb/ton

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

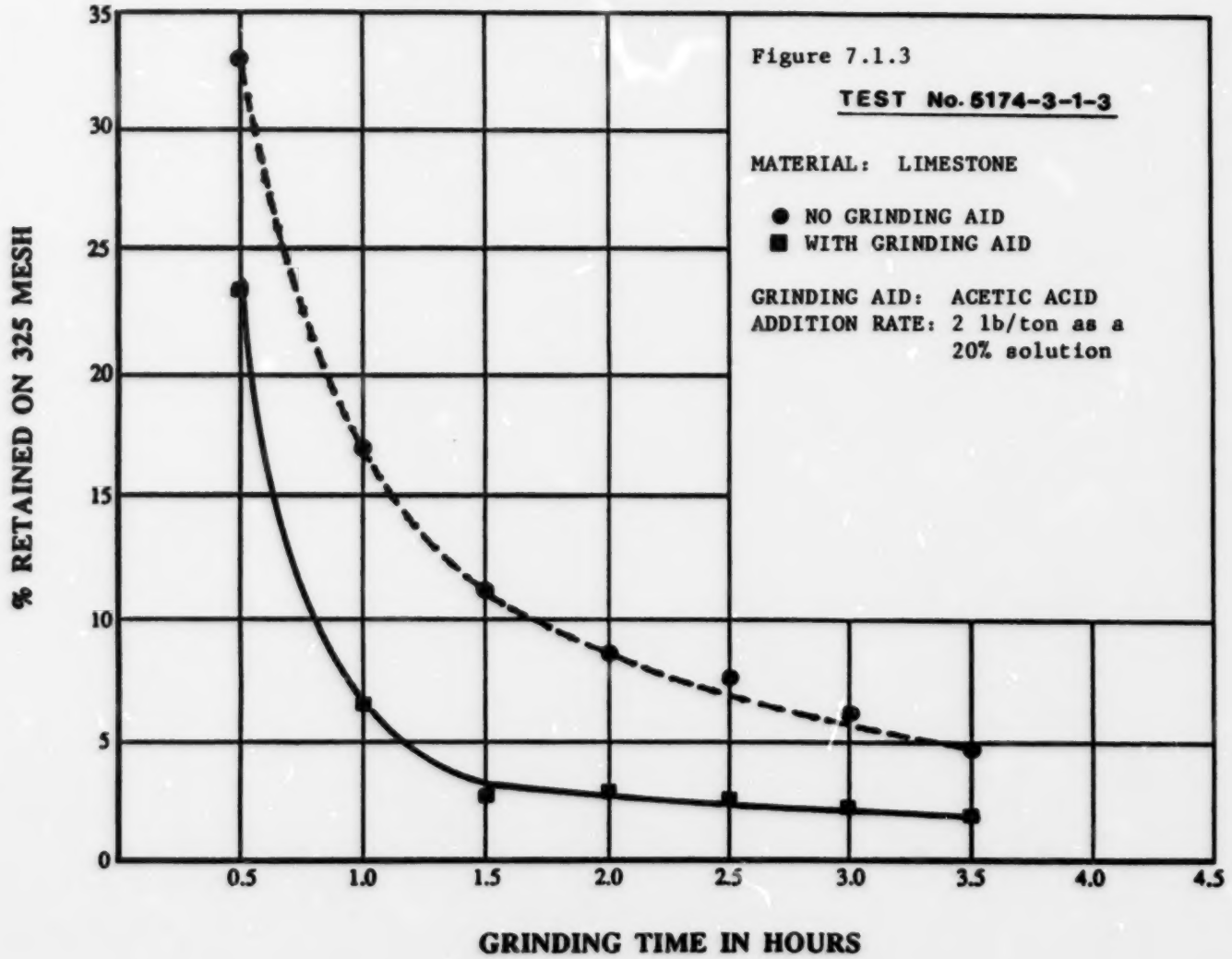


Figure 7.1.3

TEST No. 5174-3-1-3

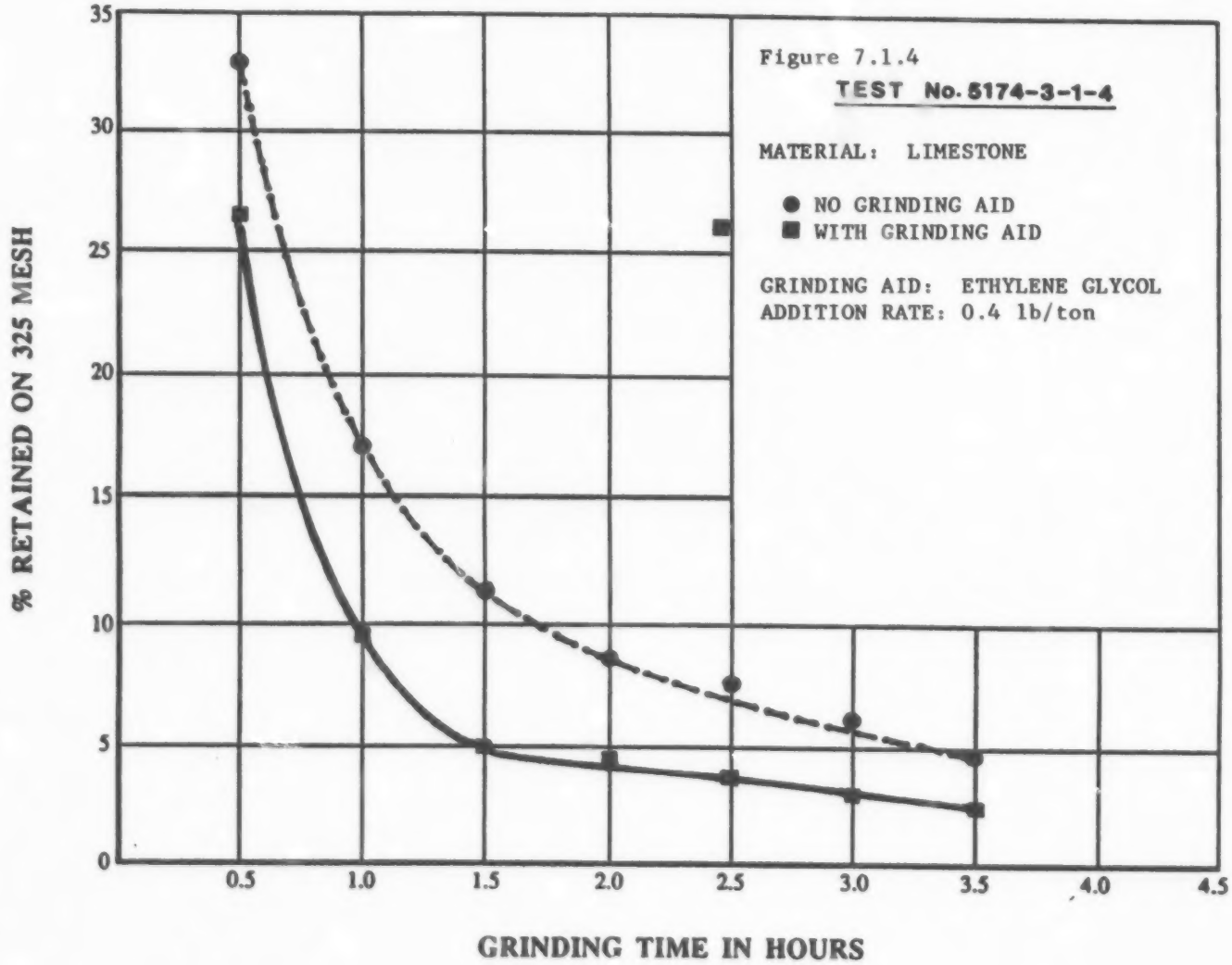
MATERIAL: LIMESTONE

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: ACETIC ACID
ADDITION RATE: 2 lb/ton as a 20% solution

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3



DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

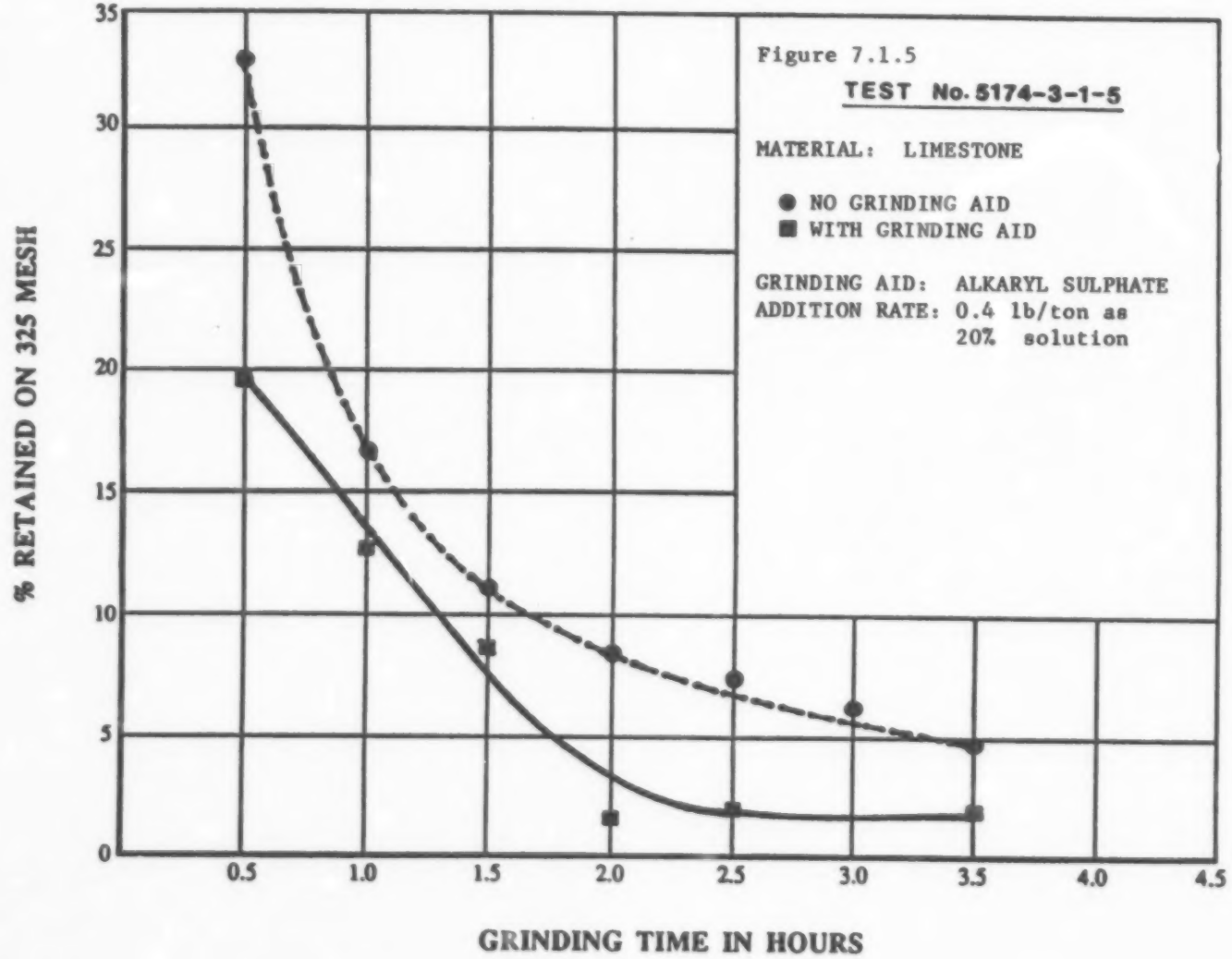


Figure 7.1.5

TEST No. 5174-3-1-5

MATERIAL: LIMESTONE

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: ALKARYL SULPHATE
ADDITION RATE: 0.4 lb/ton as 20% solution

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

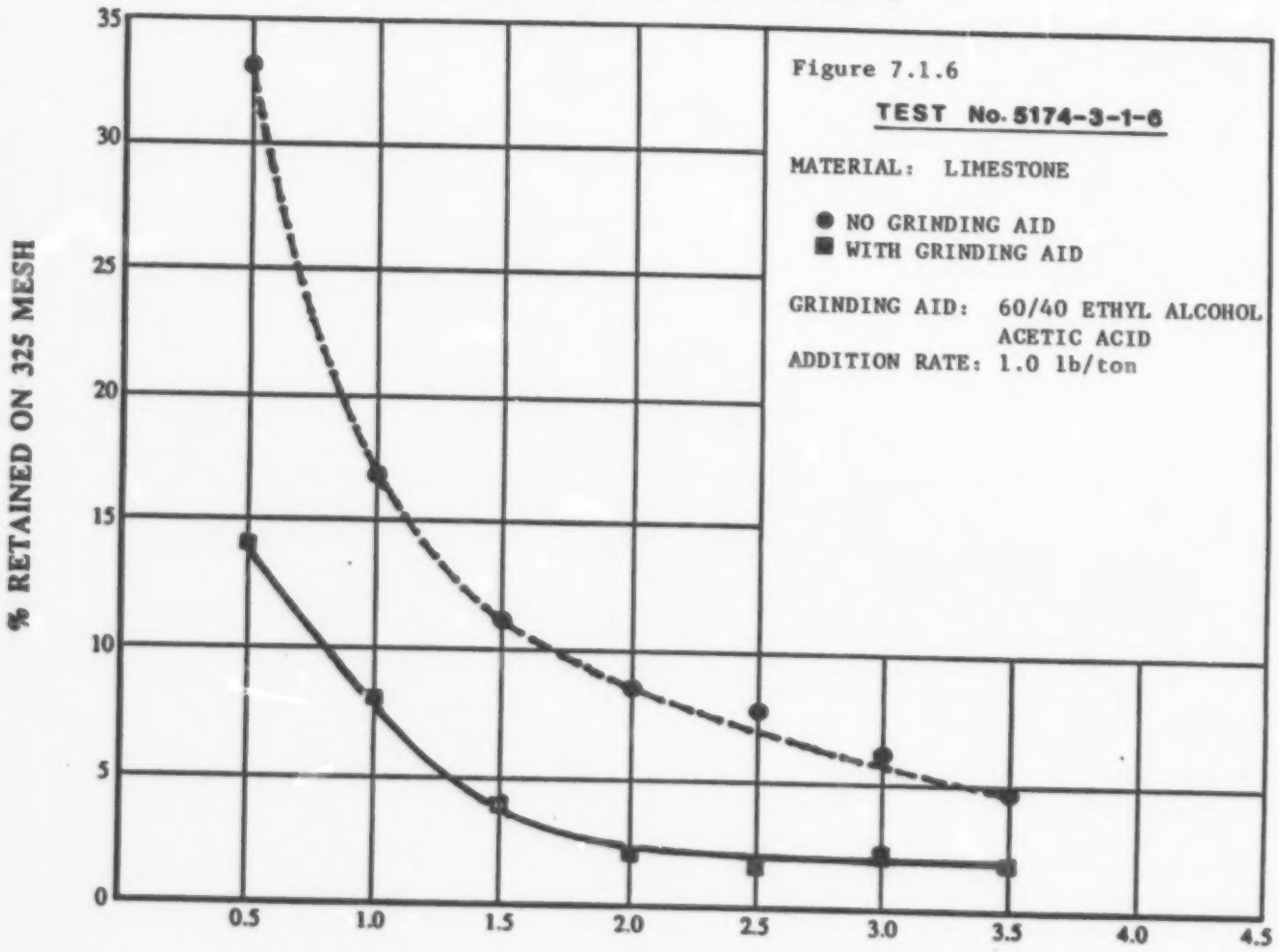


Figure 7.1.6

TEST No. 5174-3-1-6

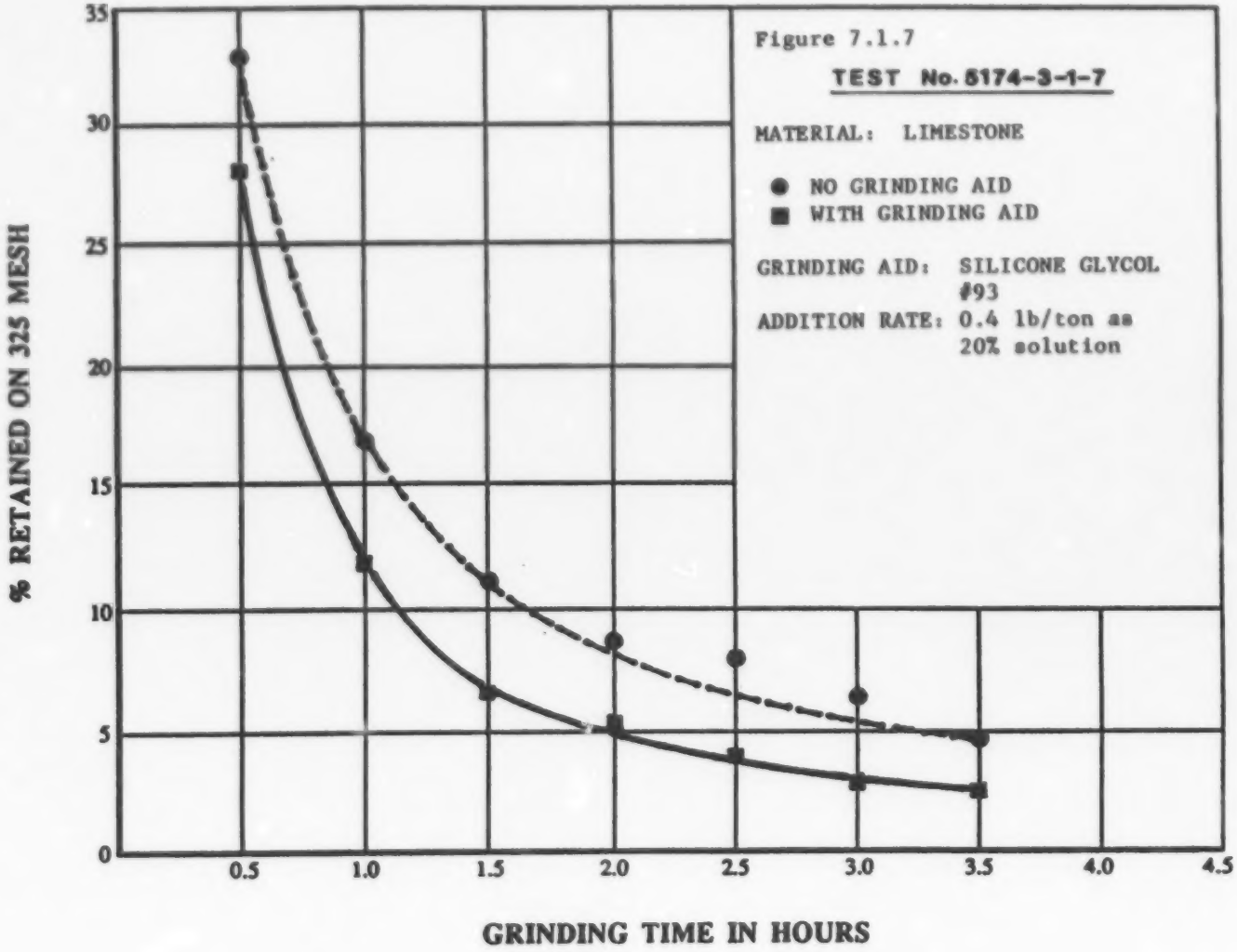
MATERIAL: LIMESTONE

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: 60/40 ETHYL ALCOHOL ACETIC ACID
ADDITION RATE: 1.0 lb/ton

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3



DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

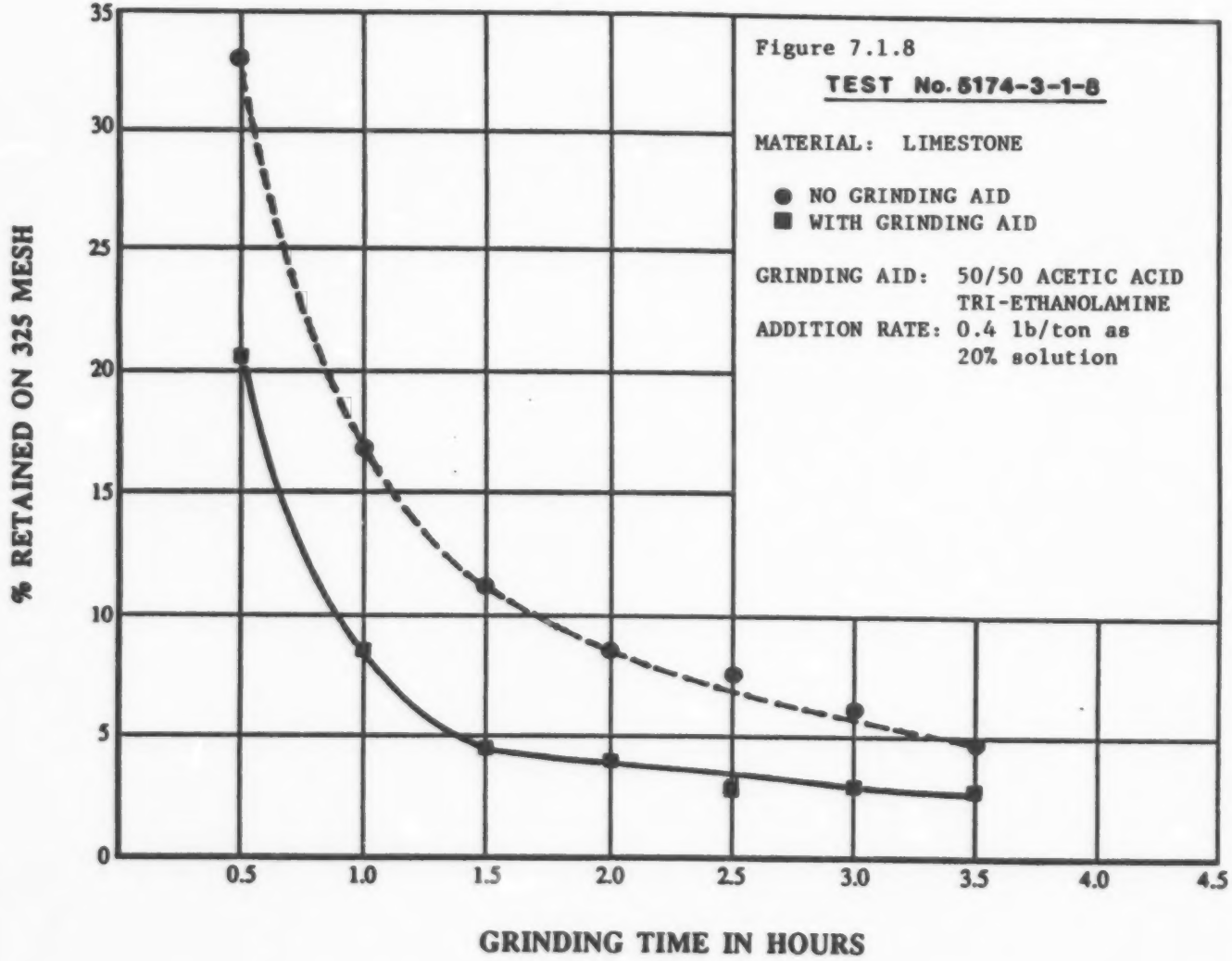


Figure 7.1.8

TEST No. 5174-3-1-8

MATERIAL: LIMESTONE

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: 50/50 ACETIC ACID
TRI-ETHANOLAMINE
ADDITION RATE: 0.4 lb/ton as
20% solution

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

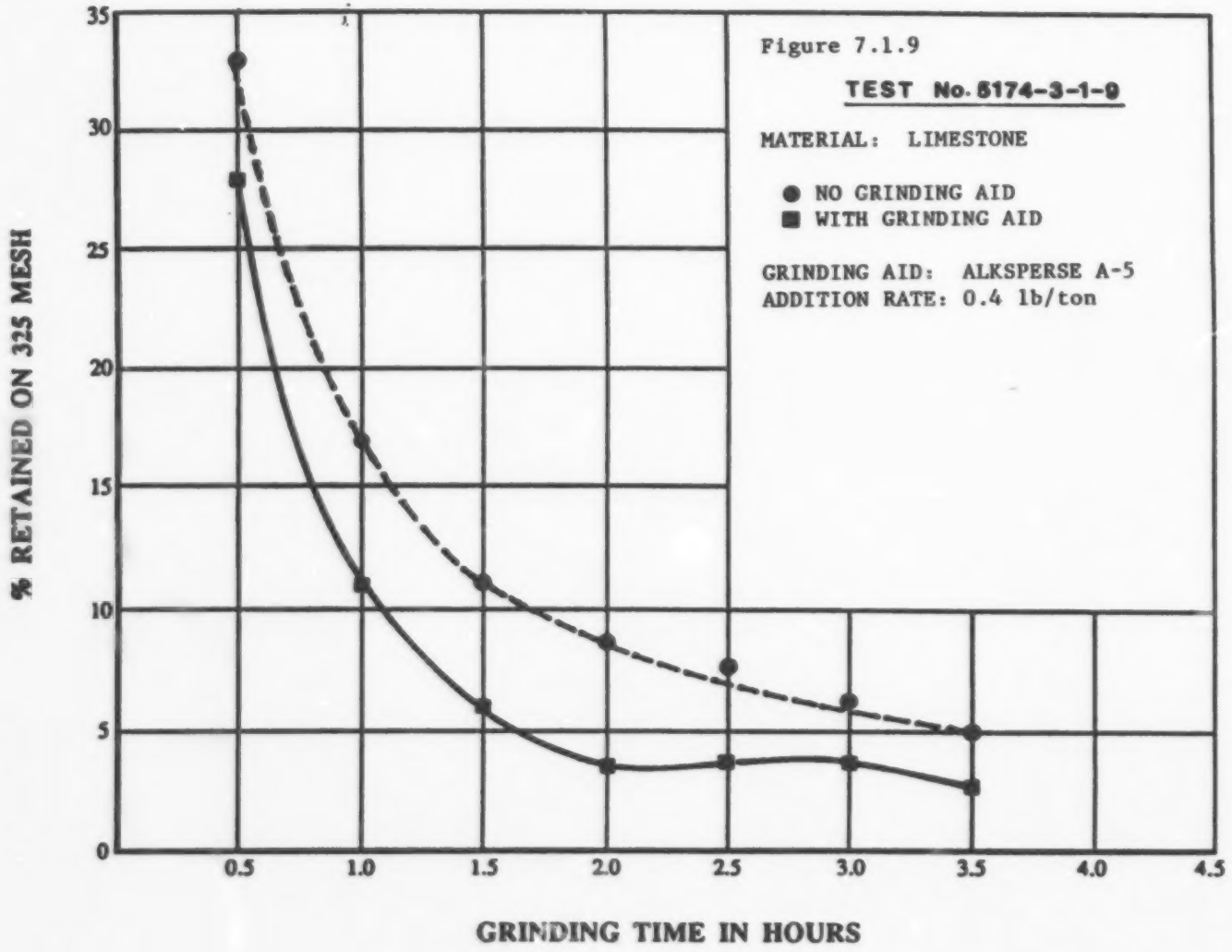


Figure 7.1.9

TEST No. 5174-3-1-9

MATERIAL: LIMESTONE

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: ALKSPERSE A-5
ADDITION RATE: 0.4 lb/ton

8.0 GRINDING OF QUARTZITE WITH CHEMICAL ADDITIVES

The feed material was a 30 X 150 mesh (495 X 105 micron) quartzite with a fine natural grain size.

The test procedure is as is outlined in Section 6.2. A total of six (6) additives were tested for their effectiveness as grinding aids. Selection of additives was based on additives as suggested in published literature taking into consideration the potential effect additives may have on ground products and their subsequent commercial uses.

8.1 Test Results

Results of the test are presented in table form in Table 8.1. A graphical presentation of grinding performance is shown in Figures 8.1.1 to 8.1.7.

8.2 Interpretation of Results

None of the chemical additives employed showed a significant increase in initial grinding rate, compared to the control sample. This indicates that this particular type of quartzite has no tendency to agglomerate or cause media or wall coating when grinding to approximately seventy (70) percent passing 325 mesh (45 micron). Past this point several of the additives, particularly silicone based chemicals showed a tendency to maintain a high grinding rate.

8.2.1 Control Sample

The control sample maintained an even grinding rate up to 1.5 hours, at which time the first evidence of coating on mill walls was noted. Grinding media started to coat after 2.5 hours, at which time there was also evidence that soft agglomerates started to form.

In comparison to tests with limestone, this quartzite showed much less of a tendency of mill and media coating. The grinding rate is fairly consistent up to seventy (70) percent passing 325 mesh (45 micron).

8.2.2 Tri-Ethanolamine - 0.025% By Weight

Tri-ethanolamine is not effective as a grinding aid for quartzite. The grinding rate parallels the control sample and is not significantly higher. Mill and media coating occurred at approximately the same time and to the same degree as with the control sample.

8.2.3 60/40 Ethyl Alcohol - Acetic Acid - 0.025% By Weight

This additive was not effective as a grinding aid, at either 0.025% or 0.05% by weight. Grinding rates were essentially the same as for the control sample. Mill wall and media coating was evident after 2.5 hours and the formation of soft agglomerates at 3.0 hours.

8.2.4 Dimethyl Silicone - 0.05% By Weight

Dimethyl silicone fluid is a water dilutable or soluable dimethyl silicone. This type of additive has proven to be an effective grinding aid in the fine grinding of quartz in vibratory mills.

In the test conducted with this additive no significant beneficial effect was observed until after 1.5 hours of grinding. After this point the grinding rate was maintained at a higher rate than without an additive.

There was no evidence of any mill or media coating during the test period. Material was extremely well dispersed and very fluid. It was found that the additive caused the material to become hydrophobic and as a result, was difficult to wet out in water.

The hydrophobicity is likely detrimental in many industrial applications and the use of this additive will be restricted to applications that are not sensitive to this phenomena.

8.2.5 Silicone Glycol Fluid 193 - 0.05% By Weight

Silicone glycol fluid 193 is a silicone glycol copolymer based surfactant.

This additive was equally as effective as the dimethyl silicone. It exhibited the same tendency of effective grinding at finer particle size. No evidence of mill or media coating was noted. The ground material showed good dispersion and fluidity.

This additive also resulted in a dry flowing but mildly hydrophobic material. As with the dimethyl silicone, it may restrict its utilization to industrial applications in which hydrophobicity is cause for concern.

This additive is certainly of interest in generating well dispersed powders with high flowability. It is expected that treatment with this type of additive be beneficial in achieving high air classification efficiency.

8.2.6 Tri-Ethanolamine - Acetic Acid - 0.05% By Weight

The use of this additive did not result in a significant improvement in the grinding rate. It did prevent formation of mill wall and media coating for up to 2.5 hours of grinding time and exhibited good dispersion and fluidity.

8.3 Conclusions For The Grinding of Quartzite With Chemical Additives

The additives tested did not show a significant positive effect in grinding quartzite. Beneficial effects are not evident until material was ground finer than eighty (80) percent passing 325 mesh (45 micron). The most effective additives are the silicone based chemicals. However, their use will be restricted due to the fact that they cause the ground material to become hydrophobic. These particular additives can be considered for any application that requires well dispersed powders with excellent dry flow properties.

Table 8.1

BENCH SCALE - STATIC GRINDING AID TEST - PROJECT 5174-3

MATERIAL - 30 X 150 MESH QUARTZITE

Grinding Aid	Addition Rate %/Wt	Percent Retained on 325 Mesh (45 micron) at Time 0.5 Hr	1.0 Hr	1.5 Hr	2.0 Hr	2.5 Hr	3.0 Hr	3.5 Hr
No Grinding Aid	-	73.1	55.1	36.0	22.3	12.6	7.4	4.6
Tri-ethanolamine	0.025%	72.9	51.1	33.4	16.6	6.9	3.4	2.3
60/40 Ethyl Alcohol Acetic Acid	0.025%	72.0	52.3	34.6	16.9	7.1	3.4	2.2
60/40 Ethyl Alcohol Acetic Acid	0.05%	71.5	51.4	34.9	17.6	7.1	2.9	1.4
Dimethyl Silicone	0.05%	71.3	51.4	28.9	12.0	4.3	1.4	0.6
Silicone Glycol	0.05%	68.0	50.3	32.3	12.9	4.6	1.4	0.5
Tri-ethanolomine - Acetic Acid	0.05%	74.3	55.1	34.9	17.1	7.4	3.1	1.9

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

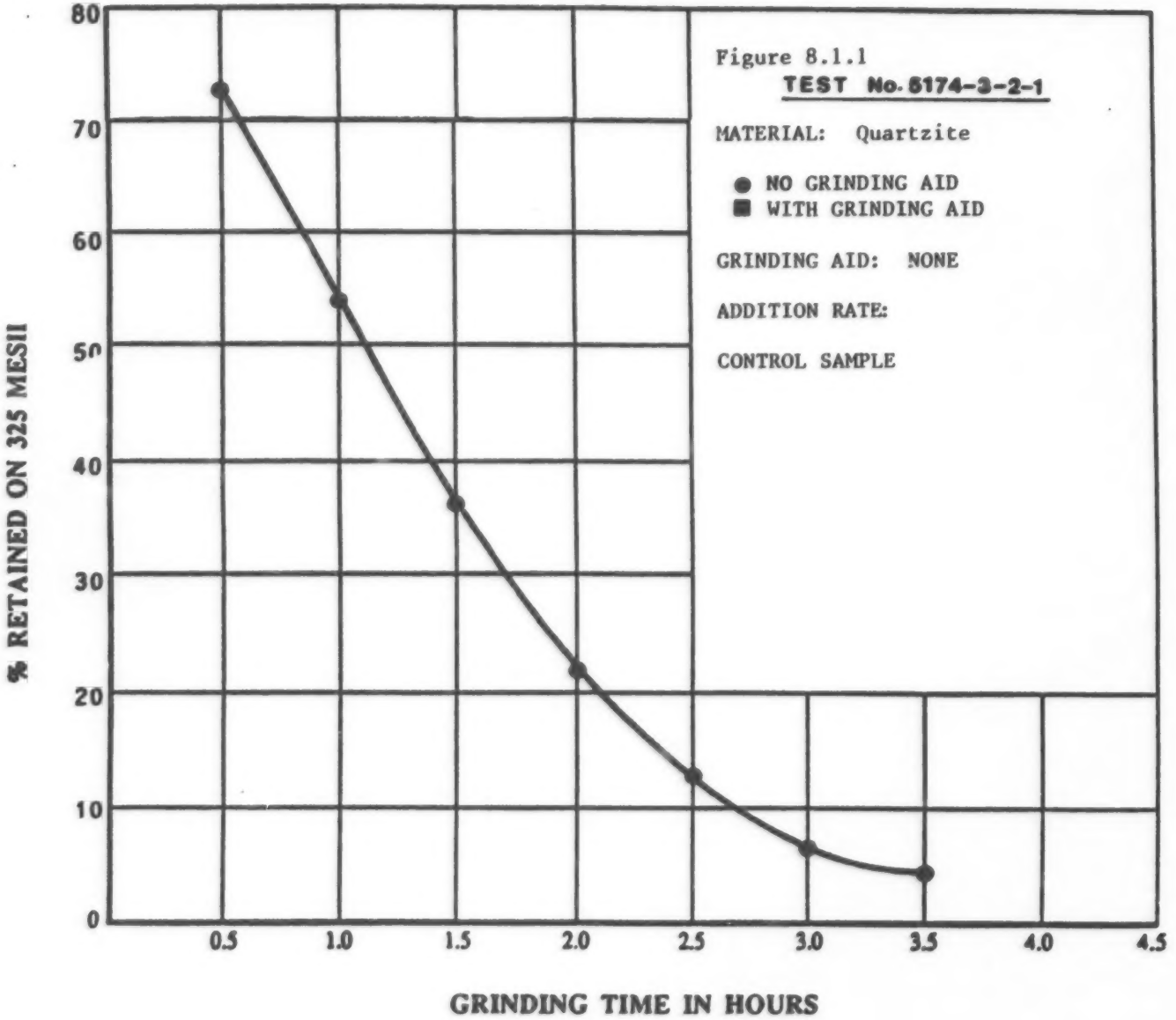


Figure 8.1.1

TEST No. 5174-3-2-1

MATERIAL: Quartzite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: NONE

ADDITION RATE:

CONTROL SAMPLE

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

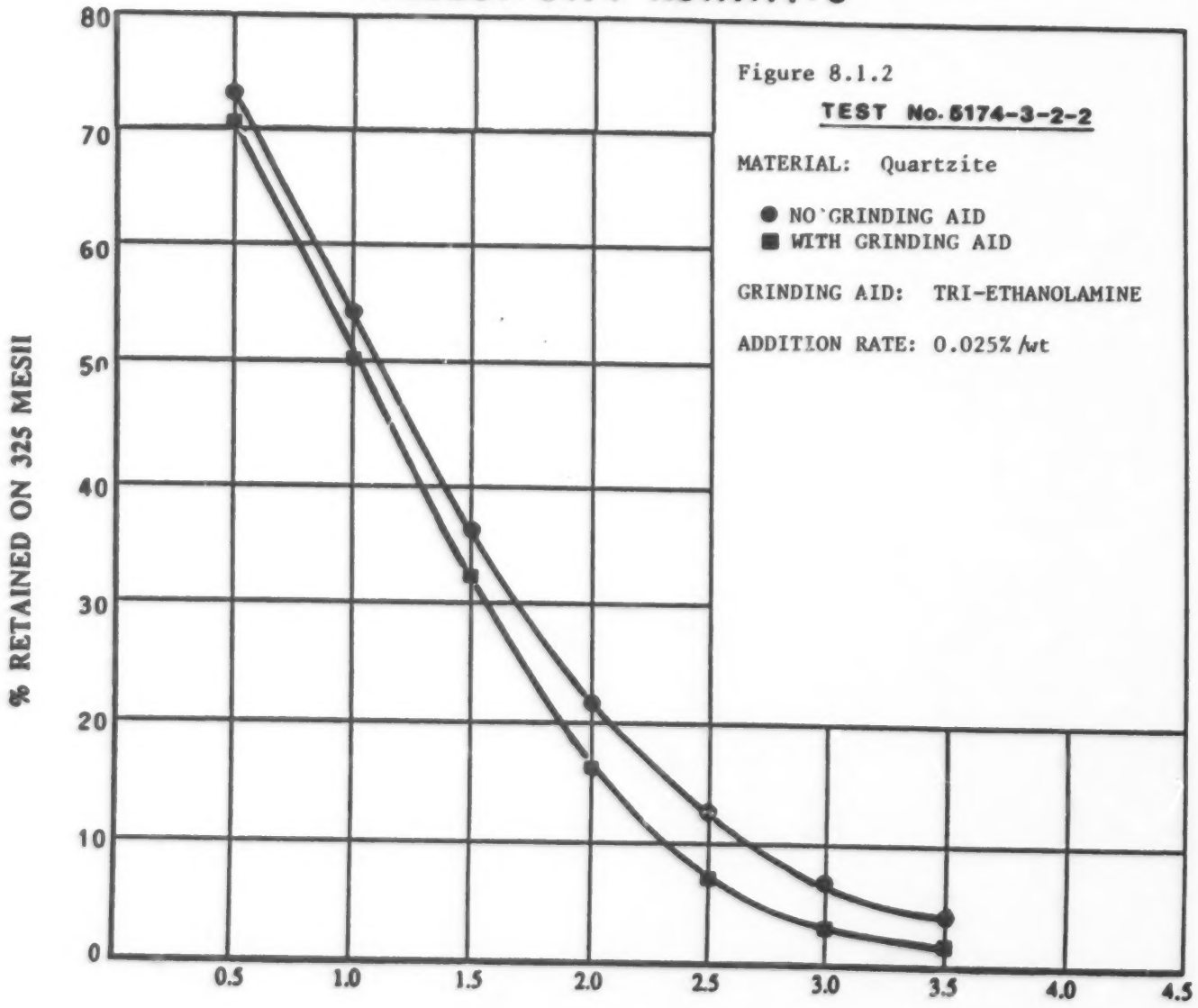


Figure 8.1.2

TEST No. 5174-3-2-2

MATERIAL: Quartzite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: TRI-ETHANOLAMINE

ADDITION RATE: 0.025% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

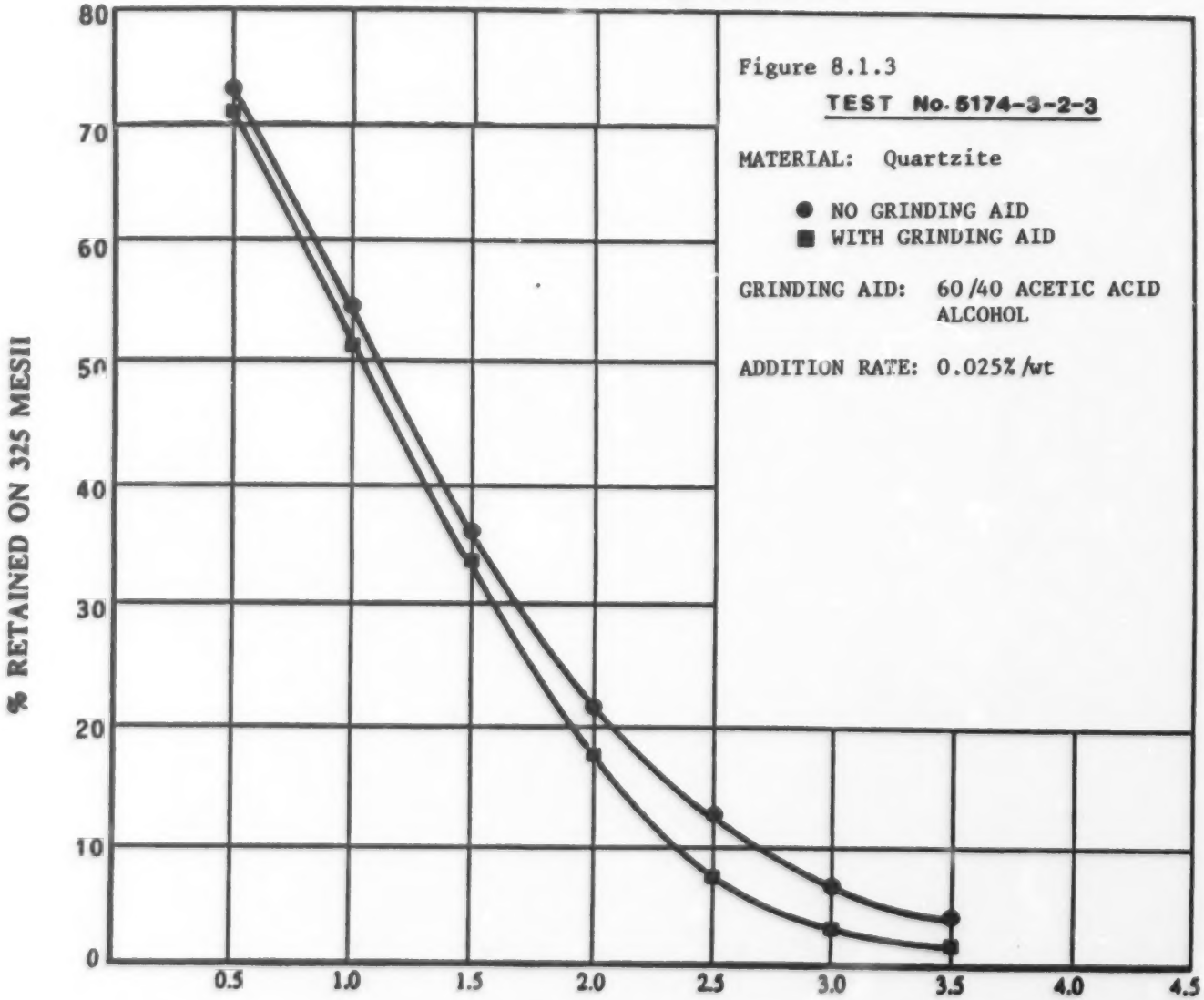


Figure 8.1.3

TEST No. 5174-3-2-3

MATERIAL: Quartzite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: 60/40 ACETIC ACID ALCOHOL

ADDITION RATE: 0.025% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

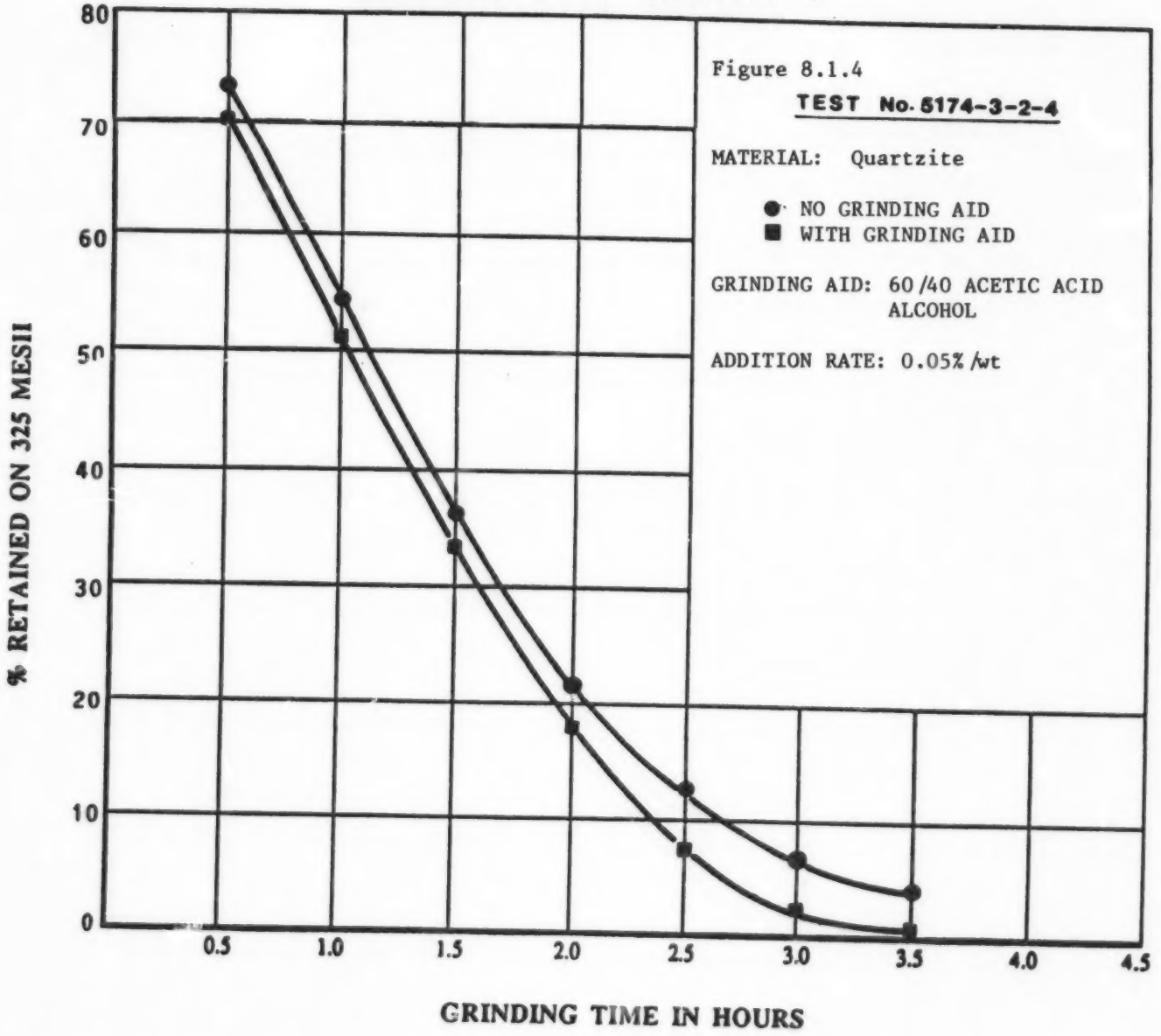


Figure 8.1.4

TEST No. 5174-3-2-4

MATERIAL: Quartzite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: 60/40 ACETIC ACID ALCOHOL

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

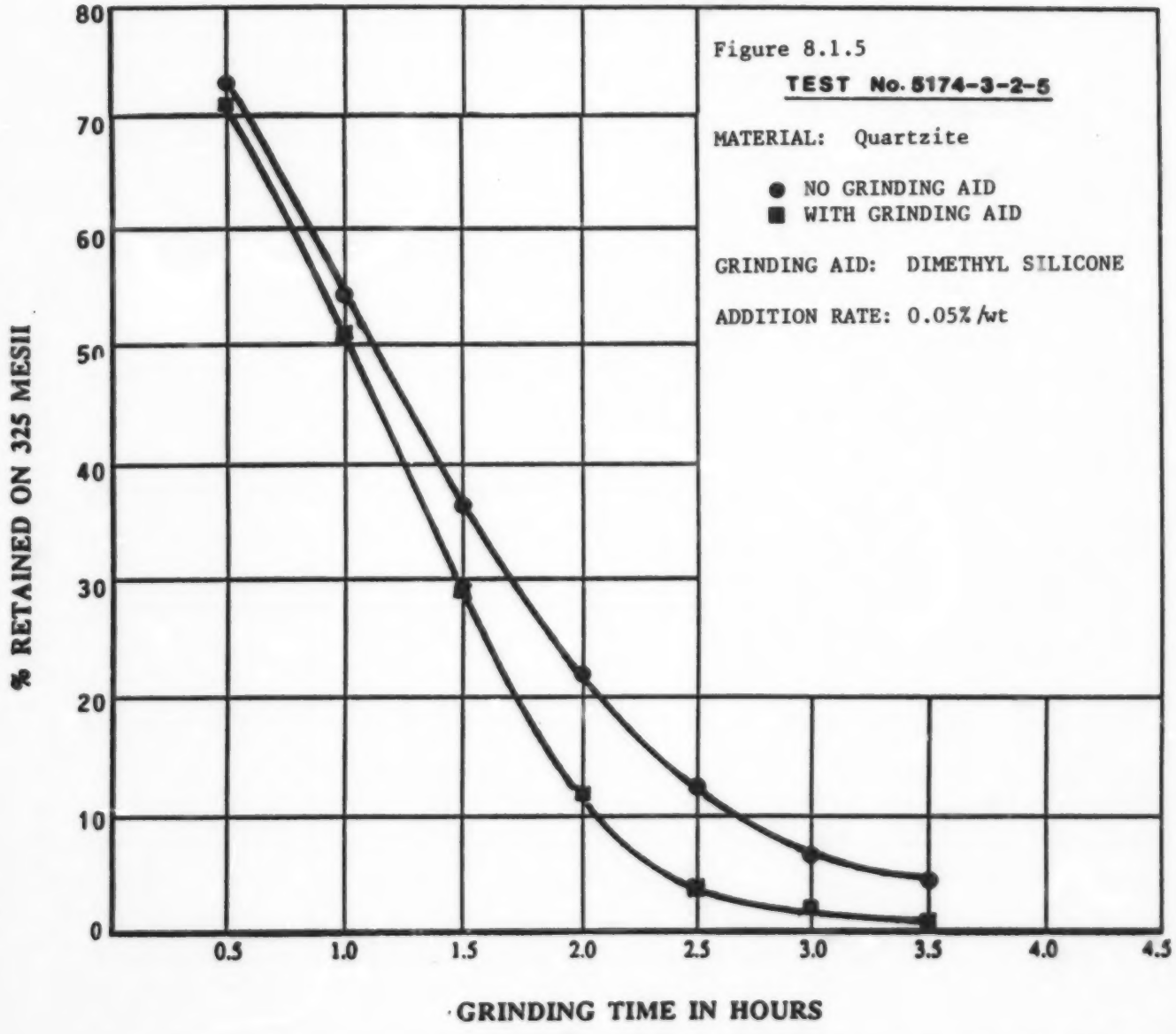


Figure 8.1.5

TEST No. 5174-3-2-5

MATERIAL: Quartzite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: DIMETHYL SILICONE

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

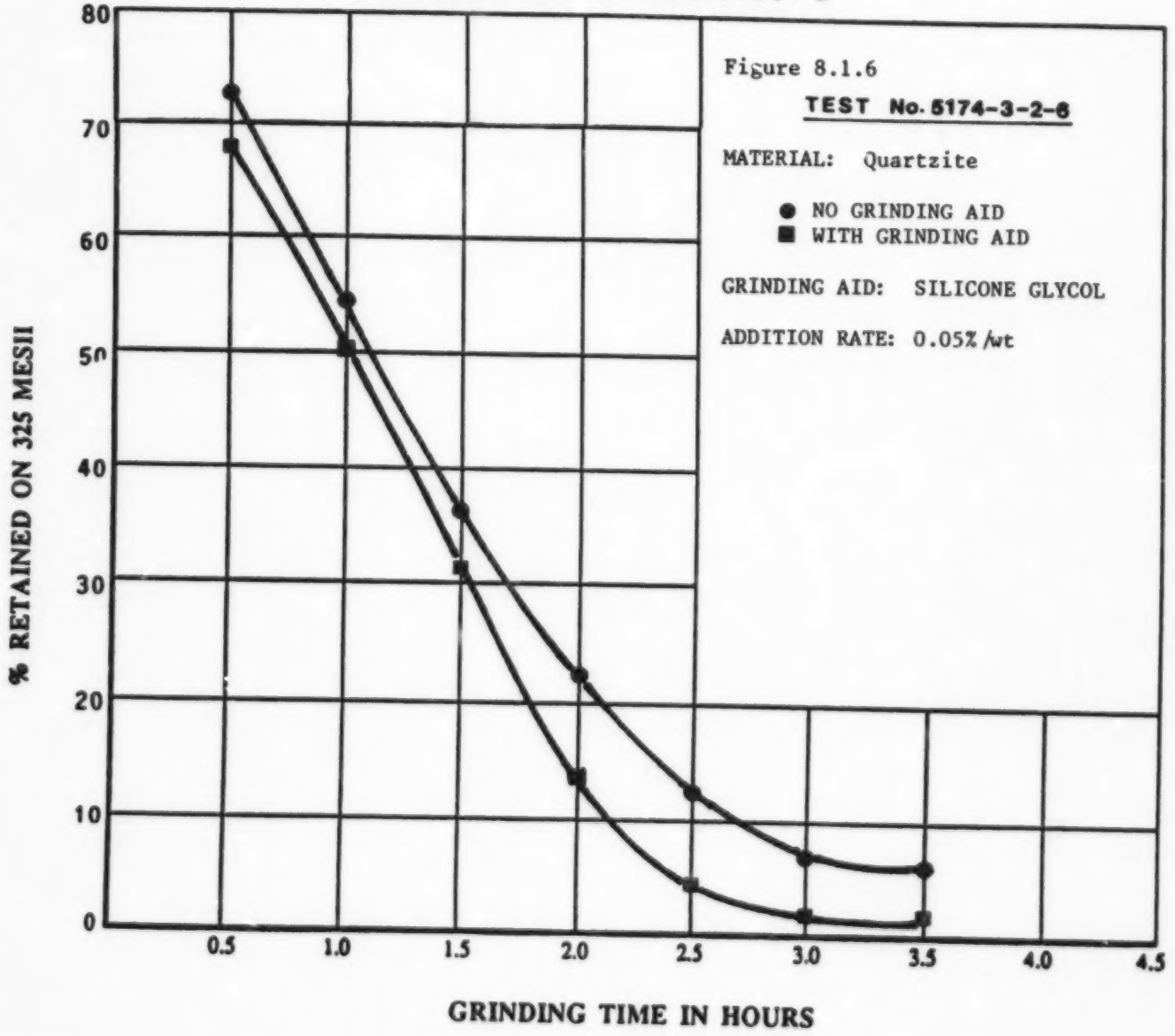


Figure 8.1.6

TEST No. 5174-3-2-6

MATERIAL: Quartzite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: SILICONE GLYCOL

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

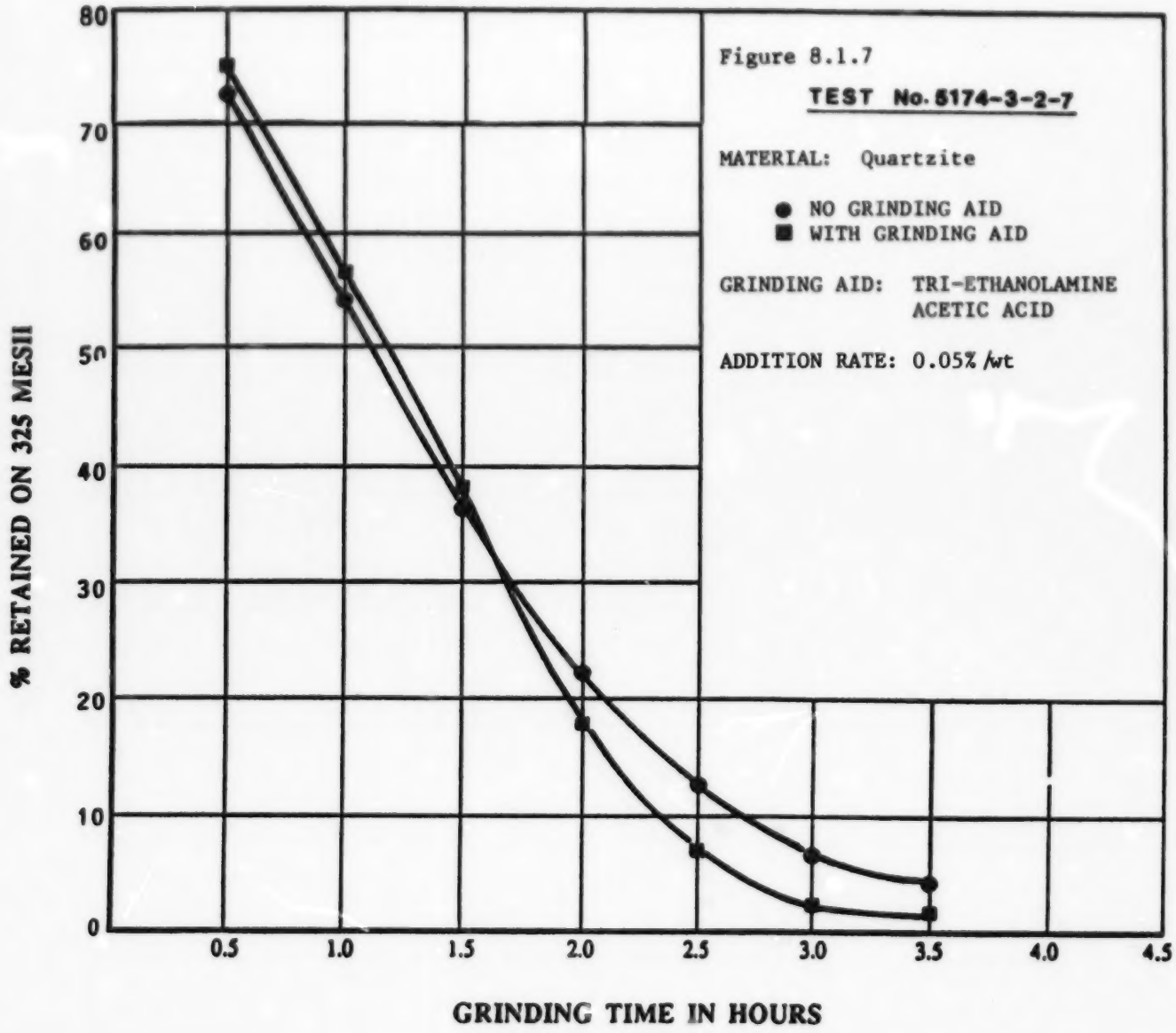


Figure 8.1.7

TEST No. 5174-3-2-7

MATERIAL: Quartzite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: TRI-ETHANOLAMINE ACETIC ACID

ADDITION RATE: 0.05% /wt

9.0 GRINDING OF NEPHELINE SYENITE WITH CHEMICAL ADDITIVES

The test material used in this test program was a 20 X 40 mesh (833 X 417 micron) nepheline syenite.

The test procedure employed was as described in Section 6.2. A total of seven (7) additives were tested for their effectiveness as a grinding aid.

Literature reviewed did not include any reference to the use of chemical additives in grinding nepheline syenite. Tests were conducted using additives commonly employed as additives in grinding other minerals or cement.

9.1 Test Results

Results of tests as conducted are given in tabular form on Table 9.1. Graphical presentation of results is given in Figure 9.1.1 to 9.1.7.

9.2 Interpretation of Results

Of the additives tests, several gave positive results. Comparing results achieved with additives with the control sample, show that improved grinding results when chemical additives are employed. In common with grinding other minerals, tri-ethanolamine gave the best overall results. A mixture of ethyl alcohol - Acetic acid also showed promise as did a silicone glycol surfactant.

Results also provide a good example of an additive that resulted in a pronounced retardation of the grinding process. The particular additive ethylene glycol gave a coarser size distribution at each cycle except the last.

9.2.1 Control Sample

In grinding the control sample, coating of the mill wall was evident after 1.5 hours. Media coating was observed after 2.0 hours and agglomeration at 3.0 hours.

9.2.2 Ethylene Glycol - 0.05% By Weight

Results with ethylene glycol were surprising. Rather than acting as a grinding aid, an actual retardation of the grinding process was observed. Samples taken at the end of each cycle consistently showed a coarser size distribution than the control sample. Mill coating was observed after 1.5 hours and media coating at 2.0 hours. Agglomeration was noted at 2.5 hours, similar to the control sample. After 3.0 hours of grinding, both the control and the sample ground with ethylene glycol had similar residue retained on a 325 mesh (45 micron) sieve. No explanation can be offered for the retardation of the grinding process.

9.2.3 Tri-Ethanolamine - 0.05% By Weight

Test results indicate that tri-ethanalomine is an effective additive in the grinding of nepheline syenite. Each of the grinding cycles resulted in products with significantly lower residues retained on a 325 mesh (45 micron) sieve.

No mill wall or media coating were observed until after 2.5 hours of grinding, indicating that this additive is effective in retarding formation of coatings therefore extending the grinding action.

Of interest is the fact that this additive showed an increased grinding rate up to 1.5 hours. This is significant since the control sample did not give any indication of a reduced grinding rate due to formation of mill or media coating for the first two grinding cycles (1 hour) and only limited mill wall coating after 1.5 hours.

Results suggest that this additive is effective, if fine grinding is required. It suggests that greater amounts of product can be produced in equal grinding time resulting in a reduction in energy requirements.

9.2.4 60/40 Ethyl Alcohol - Acetic Acid - 0.05% By Weight

This additive, like tri-ethanolamine, showed an increase in the grinding rate during for up to 1.5 hours. After this, the grinding rate slowed down. No further grinding was observed after 2.5 hours. Mill and media coating was evident at 2.0 hours. Agglomeration was noted after 2.5 hours of grinding.

This suggests that the additive can be used for grinds up to eighty (80) percent passing 325 mesh (45 micron). Beyond this point the grinding rate is reduced significantly due to mill wall and media coating. The high initial grinding rate is such that it should result in increased productivity if mill parameters can be adjusted to take advantage of this. Increased productivity would be achieved at equal energy consumption to material ground without an additive.

9.2.5 Dimethyl Silicone Fluid - 0.05% By Weight

The dimethyl silicone used is a water dilutable fluid containing 35% dimethyl silicone. This type of additive has reportedly been used in the grinding of quartz and other silicate minerals in the U.S.S.R.

Use of this additive resulted in a high initial grinding rate for the first four grinding cycles i.e. up to 2.0 hours of grinding, after which the grinding rate slowed down significantly. This coincided with the observed coating of the mill walls after 1.5 hours of grinding and coating of grinding media after 2.0 hours.

The observed high initial grinding rate is of interest as it affords the opportunity to produce larger quantities of material at equal energy consumption. This is only valid for grinds up to eighty (80) percent passing 325 mesh (45 micron). This may require some modificationn of milling conditions. The material shows good dispersion and flow characteristics, which may result in a reduction of mill residence time and an increase in recirculating loads. This may be corrected by reducing the discharge grate configuration or the open area of the discharge grates.

9.2.6 H.E.A. - 2 - 0.05% By Weight

H.E.A.-2 is a commercially available grinding aid and is an acetate form of ethanolamine. This additive is used extensively by the cement industry.

This additive showed limited effectiveness. The initial grinding rate was similar to the control sample but showed a better rate of grinding after one (1) hour. This is attributed to the fact that no mill wall coating or media coating was observed until after 2.0 hours of grinding. This indicates that H.E.A.-2 is reasonably effective in retarding the onset of coatings and/or agglomeration. The overall benefits are less than those indicated for additives such as tri-ethanolamine or alcohol-acetic acid.

9.2.7 Silicone Glycol - Surfactant 193 - 0.05% By Weight

Silicone glycol fluid 193 is a silicone glycol copolymer based surfactant. This type of chemical is considered to be a good dispersant and flow aid for silica or silicate minerals.

This additive showed a high initial grinding rate compared to the control sample, similar to the dimethyl silicone fluid. No mill wall coating or media coating was observed until after 2.0 hours. Agglomeration was evident after 3.0 hours and after 2.0 hours the grinding rate was reduced. However, the rate was significantly higher than for the control sample. The product showed good dry dispersion and flow characteristics. In actual grinding practice this may result in a reduction in mill residence time and increased circulating loads in closed circuit grinding operations.

9.3 Conclusions on Grinding Nepheline Syenite With Chemical Additives

Of the additives tested, tri-ethanolamine was the most effective and least costly. It appears to be useful over a broader range of particle size distribution compared to alcohol-acetic acid, dimethyl silicone and silicone glycol which were not as effective at fine grinds.

Only "in-plant" testing under dynamic production conditions can establish which of these additives is most suitable for the task. Milling conditions may have to be adapted or modified to achieve optimum results. It is well documented in the literature that actual milling conditions can have a significant impact on the quality of results that can be achieved. Enhancement in air classification is another factor that must be taken into account. Lack of agglomerates and/or good dry dispersion of the ground product may result in increased classifier efficiency.

Table 9.1

BENCH SCALE - STATIC GRINDING AID TEST - PROJECT 5174-3

MATERIAL - 20 X 40 NEPHELINE SYENITE

Grinding Aid	Addition Rate %/Wt	Percent Retained on 325 Mesh (45 micron) at Time 0.5 Hr	1.0 Hr	1.5 Hr	2.0 Hr	2.5 Hr	3.0 Hr	3.5 Hr
No Grinding Aid	-	72.8	54.0	38.3	24.0	15.8	10.5	7.3
Ethylene Glycol	0.05%	73.0	66.3	49.0	30.0	19.3	11.0	5.5
Tri-ethanolamine	0.05%	76.0	53.3	23.3	7.3	2.8	1.0	0.75
60/40 Ethy Alcohol Acetic Acid	0.05%	68.0	45.0	20.3	13.5	8.3	5.8	4.0
Di-Methyl Silicone 35%	0.05%	73.3	42.8	22.5	10.8	6.0	3.8	3.3
H.E.A.-2	0.05%	76.8	54.8	29.3	16.5	8.0	5.3	3.5
Silicone Glycol Surfactant	0.05%	72.8	45.3	24.3	10.8	4.5	2.3	1.5

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

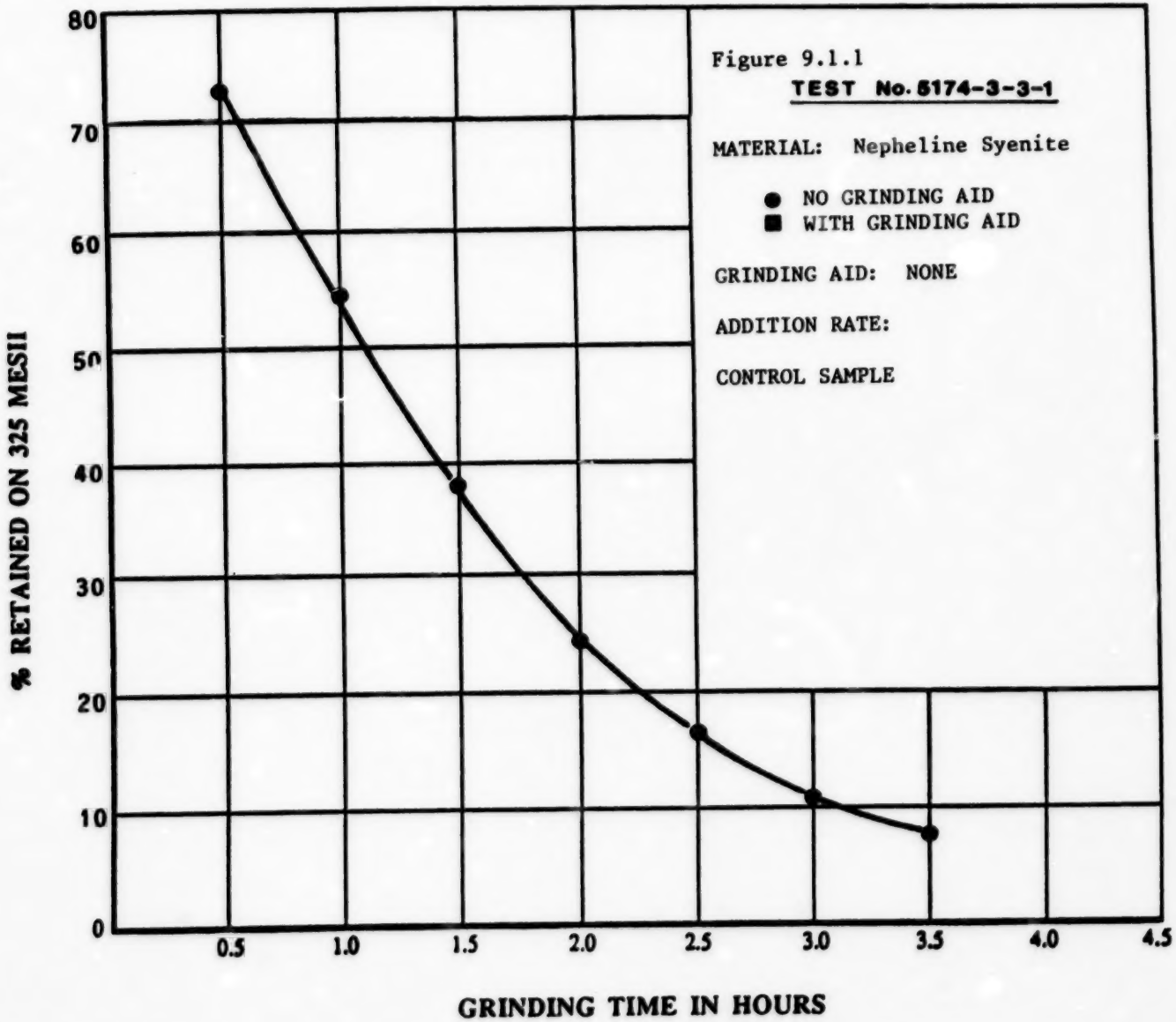


Figure 9.1.1

TEST No. 5174-3-3-1

MATERIAL: Nepheline Syenite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: NONE

ADDITION RATE:

CONTROL SAMPLE

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

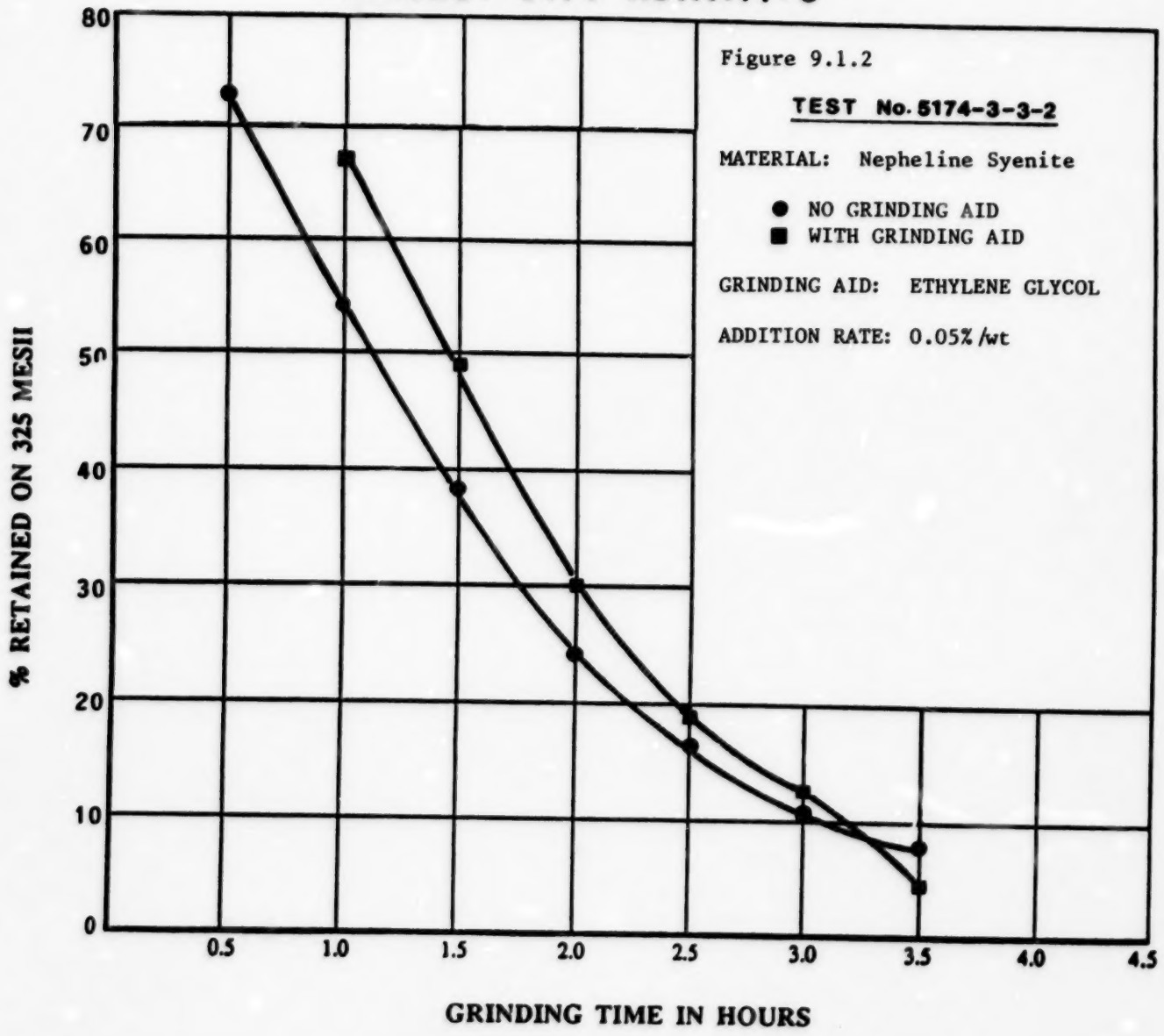


Figure 9.1.2

TEST No. 5174-3-3-2

MATERIAL: Nepheline Syenite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: ETHYLENE GLYCOL

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

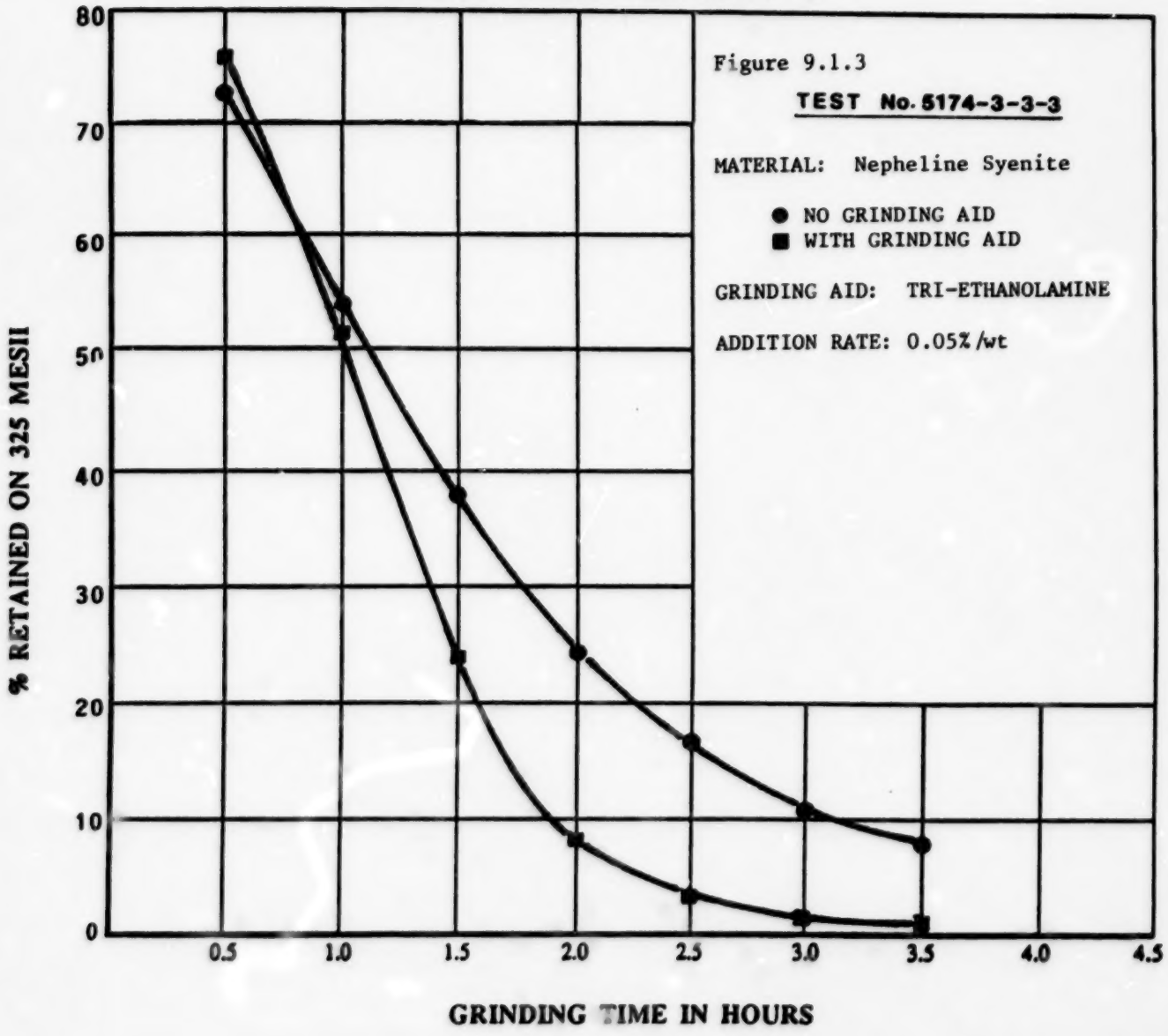


Figure 9.1.3

TEST No. 5174-3-3-3

MATERIAL: Nepheline Syenite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: TRI-ETHANOLAMINE

ADDITION RATE: 0.05%/wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

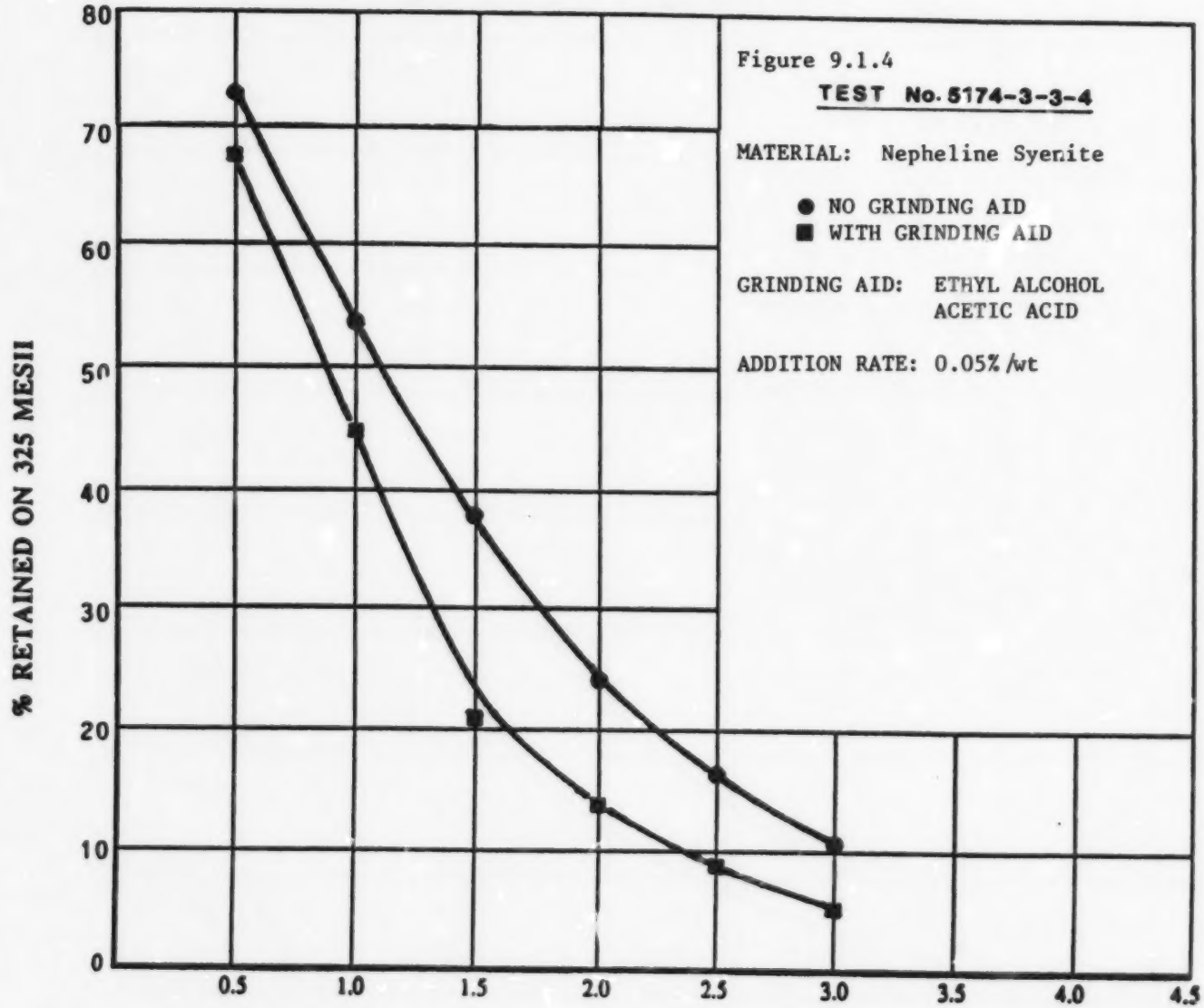


Figure 9.1.4

TEST No. 5174-3-3-4

MATERIAL: Nepheline Syenite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: ETHYL ALCOHOL
ACETIC ACID

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

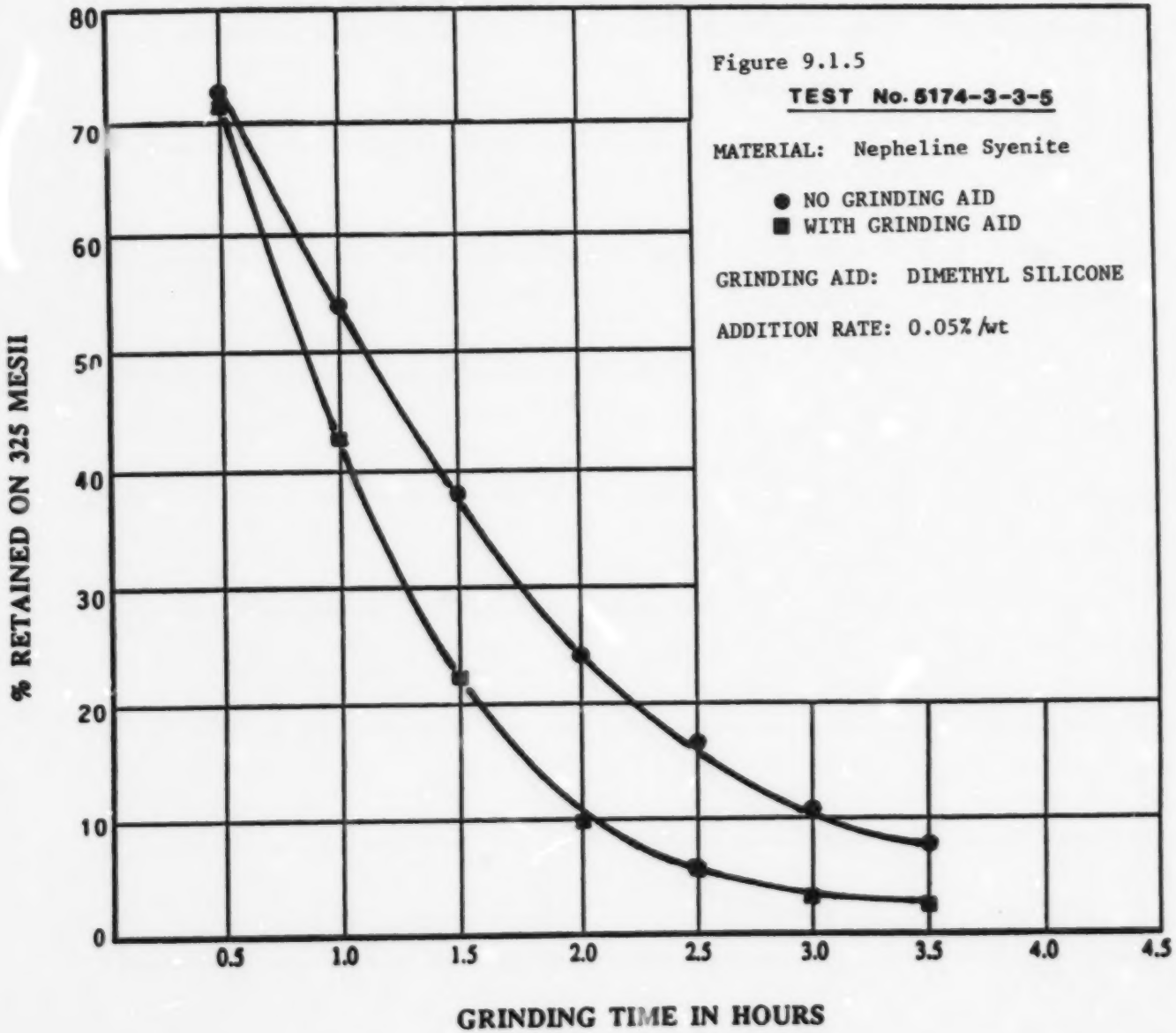


Figure 9.1.5

TEST No. 5174-3-3-5

MATERIAL: Nepheline Syenite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: DIMETHYL SILICONE

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

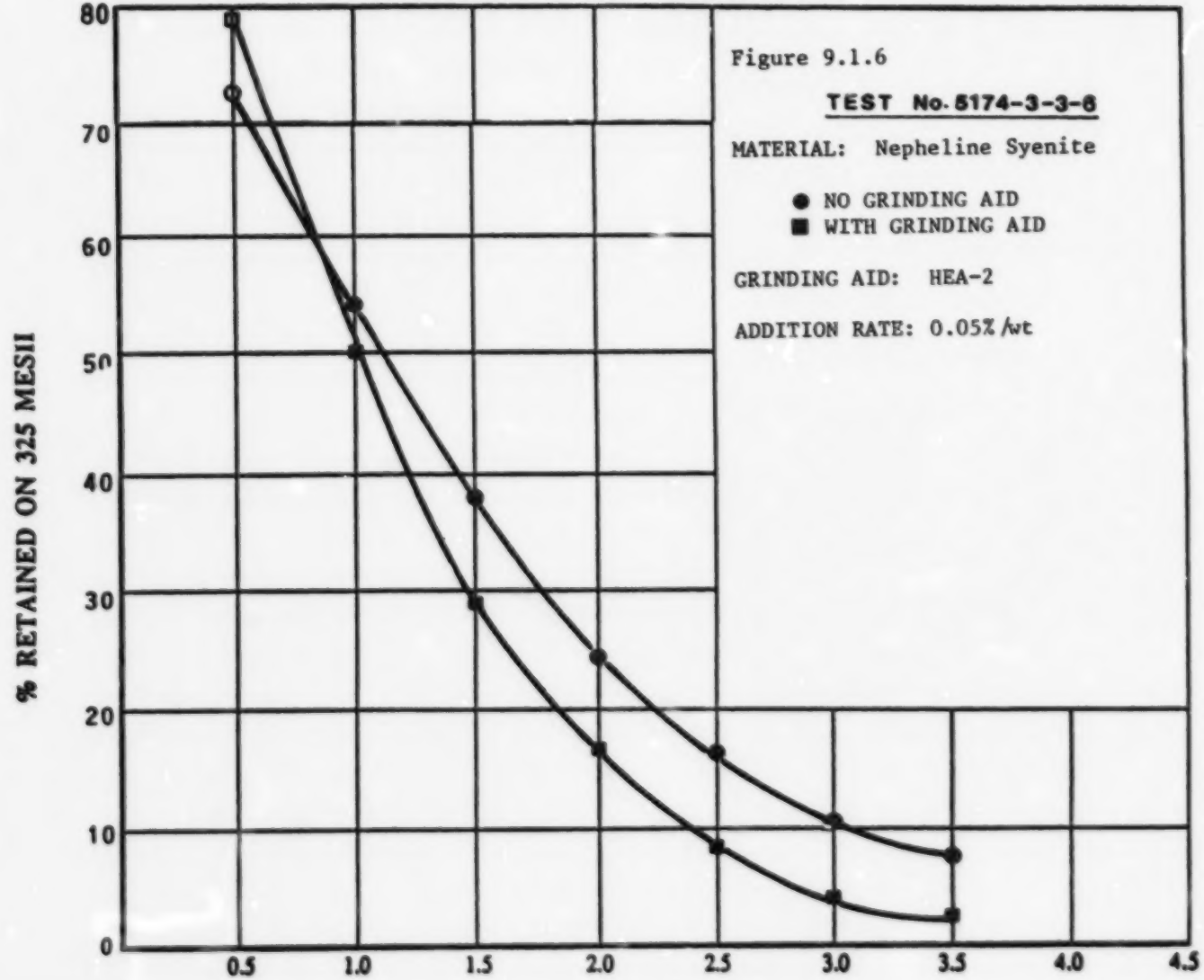


Figure 9.1.6

TEST No. 5174-3-3-6

MATERIAL: Nepheline Syenite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: HEA-2

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

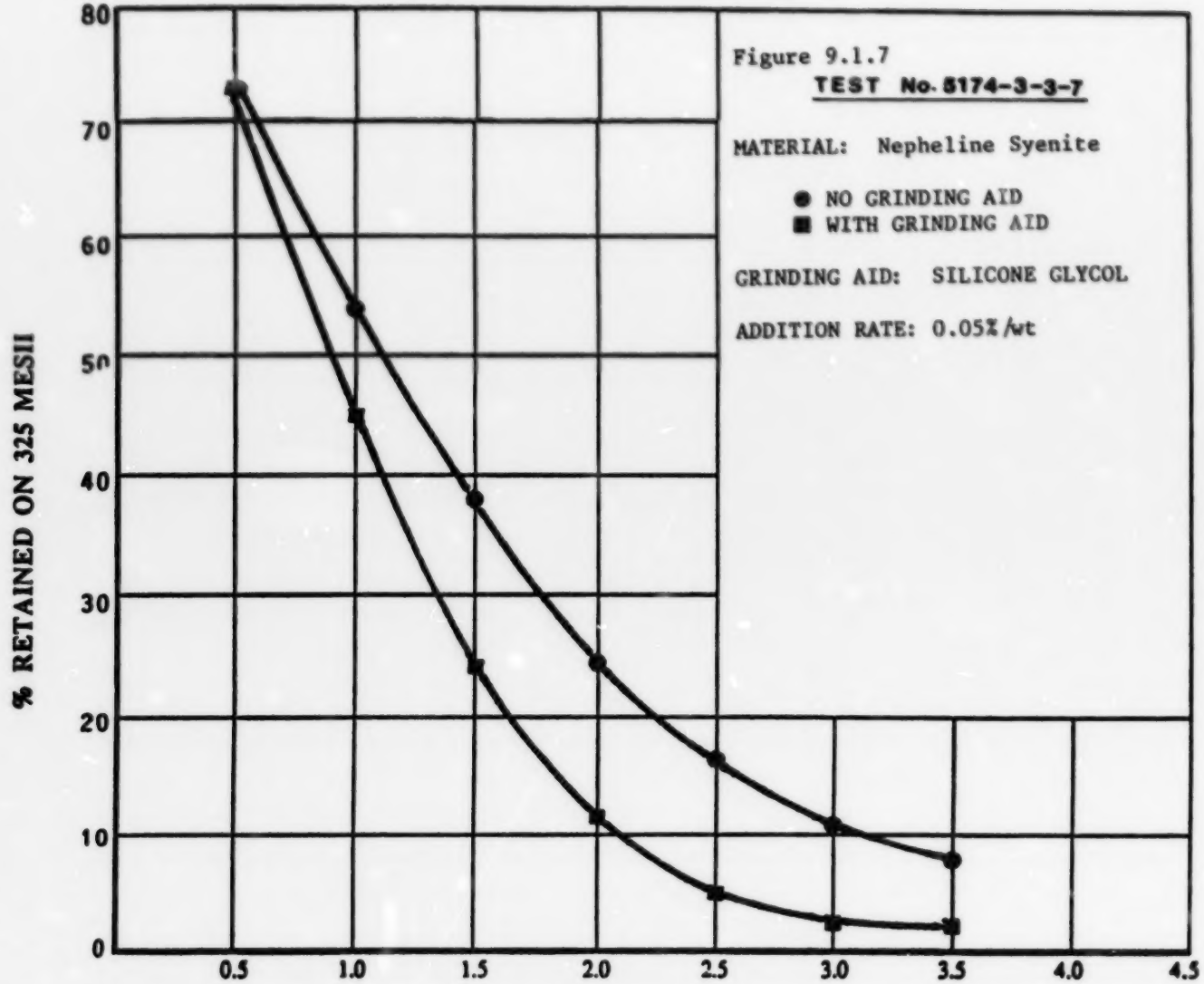


Figure 9.1.7

TEST No. 5174-3-3-7

MATERIAL: Nepheline Syenite

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: SILICONE GLYCOL

ADDITION RATE: 0.05% /wt

10.0 GRINDING OF A TALC ORE WITH CHEMICAL ADDITIVES

10.1 Results

None of seven (7) chemical additives tested resulted in improvement in grinding rates. In recently published literature, Laapas et Al[24] reported the successful use of a salt of a polycarboxylate in the dry grinding of talc in conventional tumbling mills. No specific details as to the chemical composition of this additive was disclosed.

Table 10.1 shows, in tabular form, the results as obtained in static bench scale testing. Results show no improvement of the grinding rate was achieved with any of these additives.

Table 10.1

BENCH SCALE - STATIC GRINDING AID TEST - PROJECT 5174-3

MATERIAL - 20 MESH TALC ORE 70% TALC CONTENT

Grinding Aid	Addition Rate %/Wt	Percent Retained on 325 Mesh (45 micron) at Time 0.5 Hr	1.0 Hr	1.5 Hr	2.0 Hr	2.5 Hr	3.0 Hr	3.5 Hr
No Grinding Aid	-	38.3	27.7	21.1	16.6	13.7	10.9	9.4
Tri-Ethanolamine	0.05%	25.1	21.4	16.9	13.1	10.9	8.3	6.9
60/40 Ethyl Alcohol -Acetic Acid	0.05%	34.3	26.3	15.4	12.6	11.7	8.6	7.4
Ethylene Glycol	0.05%	40.6	29.1	22.0	18.0	13.4	10.3	10.3
Silicone - Glycol	0.05%	39.7	25.4	20.3	15.7	12.3	10.0	8.2
Tri-ethanolamine -Acetic Acid	0.05%	41.1	30.0	20.3	15.7	13.4	9.4	9.7
Dimethyl - Silicone Surfactant	0.05%	42.0	28.9	22.6	17.4	14.0	11.7	9.4
H.E.A.-2	0.05%	41.7	25.7	20.9	17.7	12.3	11.3	9.4

11.0 GRINDING OF CEMENT CLINKER WITH CHEMICAL ADDITIVES

The test material used was cement clinker crushed to 100% passing 20 mesh (833 micron).

The test procedure employed is as described in Section 6.2. A total of nine (9) additives were tested. Most of these are employed extensively in the finish grinding of cement clinker.

The objectives were to evaluate grinding rates and Blaine surface area produced. Grinding times for tests with additives were based on the point at which the Blaine surface area was comparative to the Blaine surface area of the control sample ground without additive.

The Blaine surface area of the control sample was determined at a point where the residue retained on 325 mesh (45 micron) was in the order of less than ten (10) percent, in this case after 2.5 hours of grinding.

For the tests in which additives were used, the Blaine surface area was determined at the two (2) hour time interval; at which time, all but one of the samples had reached five (5) percent retained on the 325 mesh (45 micron) sieve.

11.1 Test Results

Table 11.1 gives results of the tests in tabular form. Figures 11.1.1 to 11.1.10 show, in graphical form, the progression of grinding with time.

11.2 Interpretation of Results

With the exception of a mixture of ethyl alcohol and acetic acid all of the additives tested showed a significant improvement in grinding, compared to the control sample ground without an additive. This was as expected, based on published literature on cement grinding with chemical additives.

The Blaine surface area for samples ground with additives was up to ten (10) percent higher after a 2.0 hour grinding period compared to the control sample ground after 2.5 hours.

11.2.1 Control Sample

The control sample required 2.5 hours to grind to 8.3% retained on 325 mesh (45 micron) and had a blaine surface area of 2888cm^2/gram.

Mill wall coating was noted after only one (1) hour of grinding. Grinding media coating occurred after 1.5 hours.

11.2.2 Tri-Ethanolamine - 0.025% By Weight

The test with a 0.025% addition of tri-ethanolamine showed a significantly higher grinding rate than the control sample. No mill coating or media coating was observed until 1.5 hours of grinding by which time the required particle size distribution had almost been achieved. As a result, the product after 2.0 hours of grinding had a higher Blaine surface area (3081 cm^2/gram) than the control sample after 2.5 hours.

11.2.3 60/40 Ethyl Alcohol - Acetic Acid - 0.05% By Weight

This additive was the least effective of the additives tested. It exhibited a slower initial rate of grinding and had a higher residue retained on 325 mesh (45 micron) than any of the other samples. The Blaine surface area after 2.0 hours was (3081 cm^2/gram) still significantly larger than the control sample after 2.5 hours. No mill, wall or grinding media coating was observed until after 1.5 hours of grinding time.

11.2.4 Ethylene Glycol - 0.05% By Weight

This additive used at 0.05% by weight was quite effective, showing a high initial grinding rate. No coating of mill walls or grinding media was noted until after 1.5 hours of grinding. The product after 2.0 hours of grinding had the highest blaine surface area (3154 cm^2/gram) of all the additives tested.

11.2.5 Acetic Acid - Tri-Ethanolamine - 0.05% By Weight

The grinding test with this chemical additive indicated a good initial grinding rate. No coating or agglomeration was noted during the first 1.5 hours of grinding. After 2.0 hours of grinding the product had 2% retained on 325 mesh (45 micron) and a Blaine surface area of 3024 cm^2/gram.

11.2.6 H.E.A.-2 - 0.05% By Weight

H.E.A.-2 is a commercially available grinding additive used by cement producers. The grinding test indicated a good initial grinding rate with no sticking, coating or agglomeration noted until after 1.5 hours of grinding. The Blaine surface area was 3106 cm^2/gram after 2.0 hours of grinding which was the second highest of all the additives tested.

11.2.7 M.T.D.A. - 0.05% By Weight

Like many of the additives tested in this program, M.T.D.A is an additive specifically developed for use in the finish grinding of cement. After 2.0 hours of grinding the product had the lowest percentage retained (1.4%) on 325 mesh (45 micron) of all the additives tested. Unexplainably, it had the lowest Blaine surface area (2841 cm^2/gram), which is slightly less than the control sample after 2.5 hours of grinding.

11.2.8 L.G.A. 2.2 - 0.05% By Weight

This additive is also a commercially available grinding additive, used in the finish grinding of cement. Tests indicated a high initial grinding rate. No coating or agglomeration occurred until after 1.5 hours of grinding. After 2.0 hours the product had a residue retained on 325 mesh (45 micron) of 2.6% and a Blaine surface area of 2969 cm^2/gram.

11.2.9 D.D.A.-7 - 0.05% By Weight

D.D.A. is a commercial product used in finish grinding of cement. Material ground showed a strong initial grinding rate with no coating or agglomeration observed until after 1.5 hours of grinding. The 2.0 hour ground product had a low residue (1.7%) retained on 325 mesh (45 micron) and a Blaine surface area of 2961 cm^2/gram.

11.2.10 GRAID 1 - 0.05% By Weight

This commercial grinding additive has been specifically developed for use in the finish grinding of cement. Tests with this additive showed a high initial grinding rate with no coating observed until after 1.5 hours of grinding. After 2.0 hours of grinding the product had a residue of 2.3% retained on 325 mesh (45 micron) and a Blaine surface area of 2995 cm^2/gram.

11.3 Conclusions on The Grinding of Cement With Chemical Additives

Results of the test conducted primarily with chemical additives specifically developed for the grinding of cement show conclusively the benefits that can be achieved. In all but one case it was possible to achieve a finer particle size distribution and higher blaine surface area in 2.0 hours of grinding as compared to the same clinker ground for 2.5 hours without an additive. This is equivalent to a potential productivity increase of up to twenty five (25) percent with the same energy consumption.

Results may have varied if other types of cement had been used. This explains the fact that different cement manufacturers utilize different additives in their grinding operations. Selection of an additive is a function of the cement to be ground, the fineness required and the size or type of grinding mills employed.

Other than viewing chemical additives as a grinding aid, they also contribute significantly to a reduction of pack set, better handling (conveying) characteristics and improved strength properties in cement products.

The cement industry has been a leader in the development and utilization of chemical additives, which has contributed significantly to improved energy efficiency and productivity.

Not surprisingly, most of the published literature on the use of chemical additives is as a result of the research and development conducted by the cement industry and the producers of chemical additives.

With the lead as taken by the cement industry in this particular aspect of improving dry grinding and energy conservation, producers of other industrial minerals are encouraged to follow the lead and evaluate the potential of improving the effectiveness of their grinding operations.

BENCH SCALE - STATIC GRINDING AID TEST - PROJECT 5174-3

MATERIAL - 20 MESH - CEMENT CLINKER

Grinding Aid	Addition Rate %/Wt	Percent Retained on 325 Mesh (45 micron) at Time 0.5 Hr	1.0 Hr	1.5 Hr	2.0 Hr	2.5 Hr	3.0 Hr	Blaine cm^2/gram
No Grinding Aid	-	54.0	33.7	24.9	15.7	8.3	5.7	2888*
Tri-ethanolamine	0.25%	45.4	22.3	7.7	1.7			3081
60/40 Ethyl Alcohol Acetic Acid	0.05%	51.4	32.3	17.1	7.4			3053
Ethylene Glycol	0.05%	47.4	25.1	8.6	4.0			3154
Acetic Acid Tri-ethanolamine	0.05%	47.4	25.4	9.7	2.0			3024
H.E.A.-2	0.05%	48.6	26.6	10.0	2.0			3106
MTDA	0.05%	49.7	23.1	6.9	1.4			2841
LGA 2.2	0.05%	47.4	22.9	8.9	2.6			2969
DDA-7	0.05%	45.1	21.7	7.4	1.7			2961
GRAID-1	0.05%	47.1	20.9	7.7	2.3			2995

*No grinding aid test Blaine after 2.5 hours. All others after 2.0 hours.

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

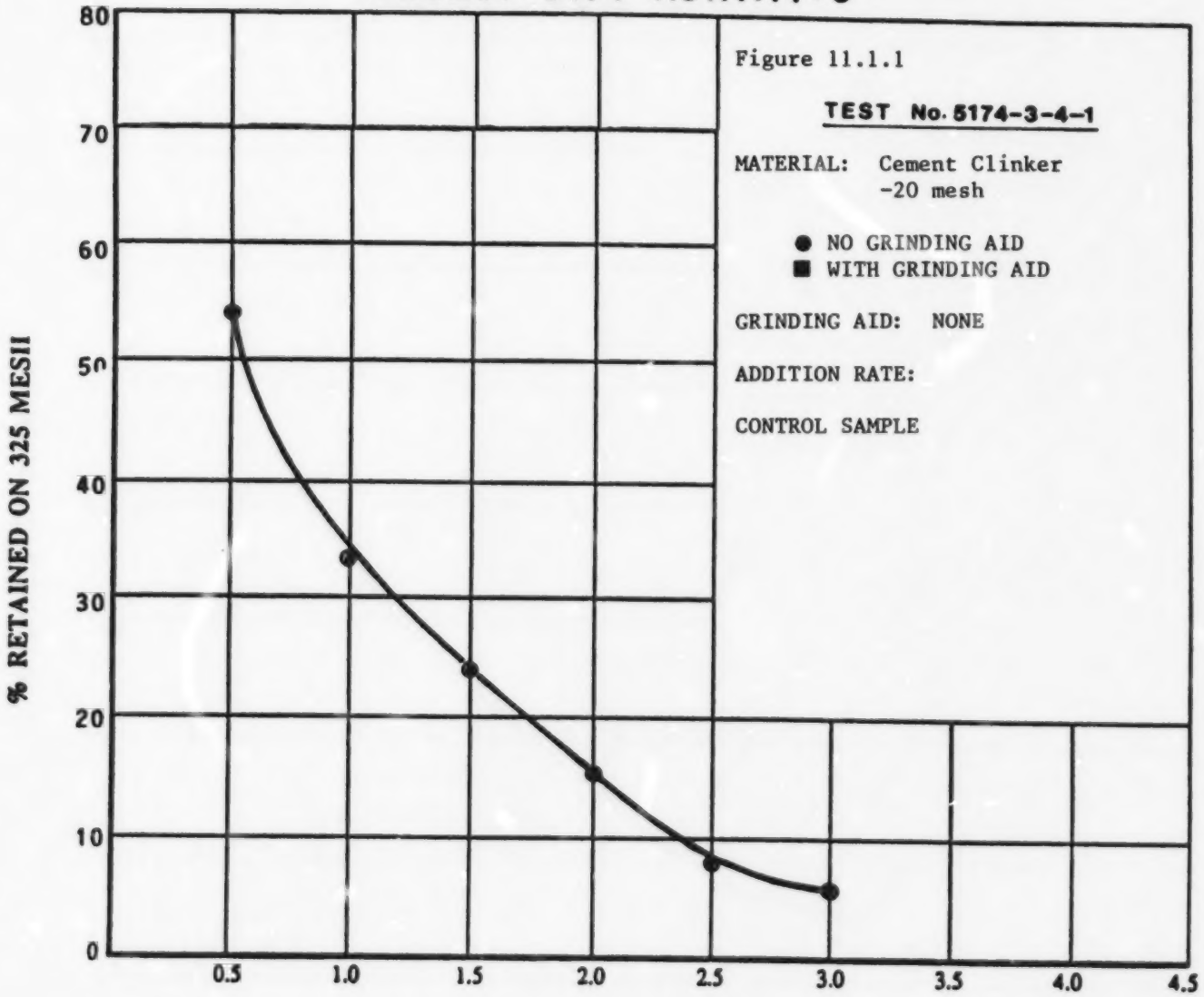


Figure 11.1.1

TEST No. 5174-3-4-1

MATERIAL: Cement Clinker
-20 mesh

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: NONE

ADDITION RATE:

CONTROL SAMPLE

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

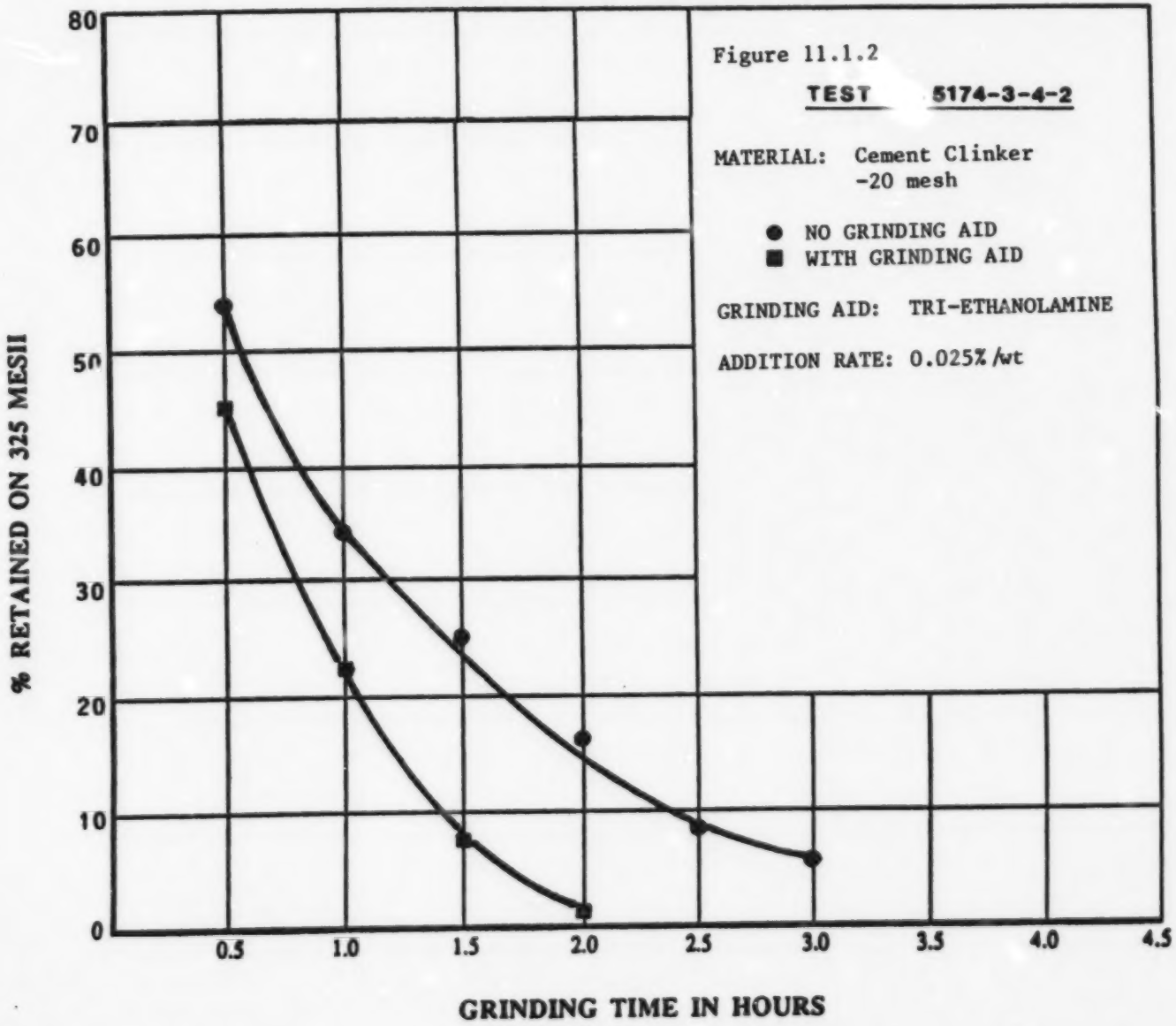


Figure 11.1.2

TEST 5174-3-4-2

MATERIAL: Cement Clinker
-20 mesh

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: TRI-ETHANOLAMINE

ADDITION RATE: 0.025% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

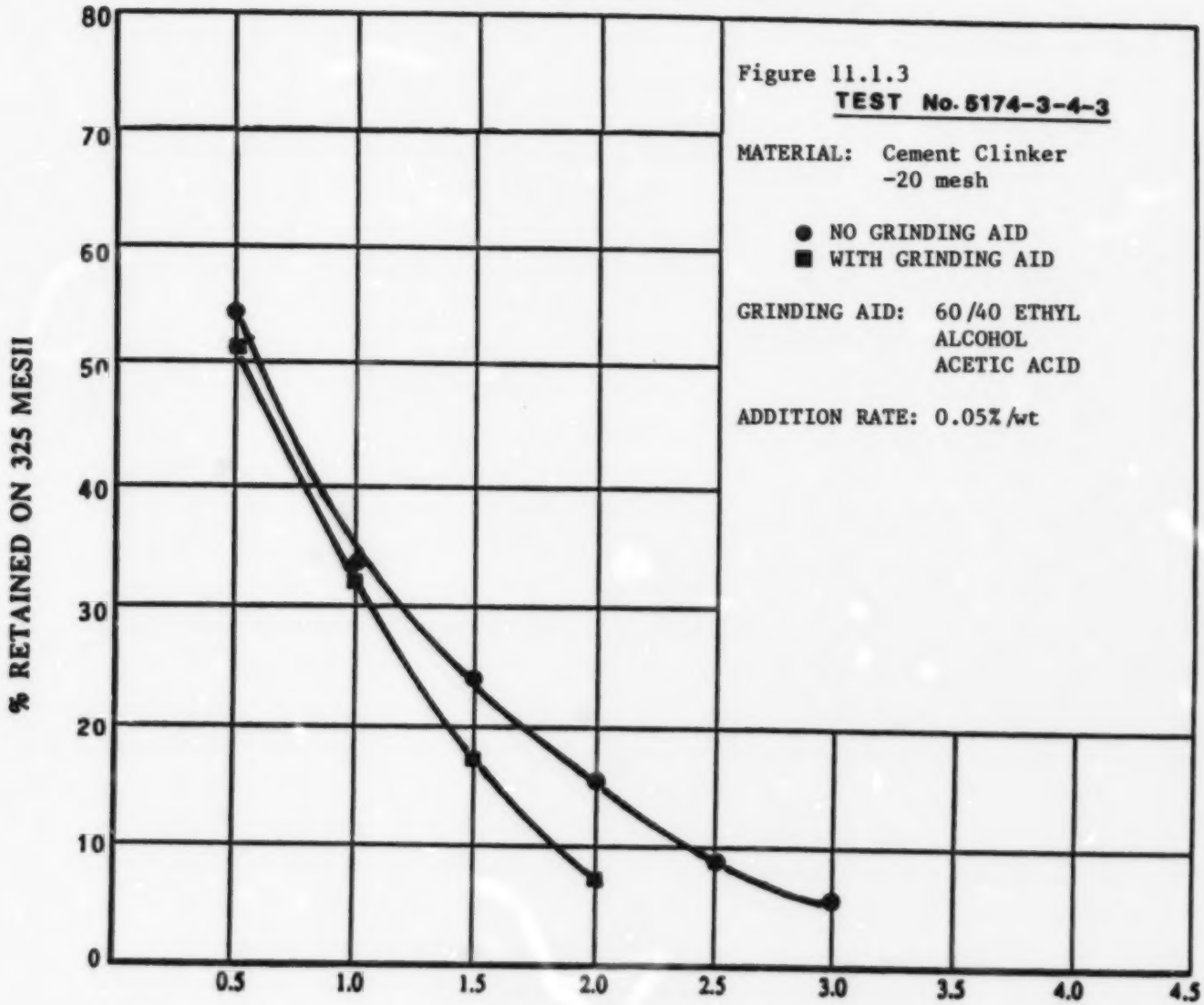


Figure 11.1.3

TEST No. 5174-3-4-3

MATERIAL: Cement Clinker
-20 mesh

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: 60/40 ETHYL ALCOHOL ACETIC ACID

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

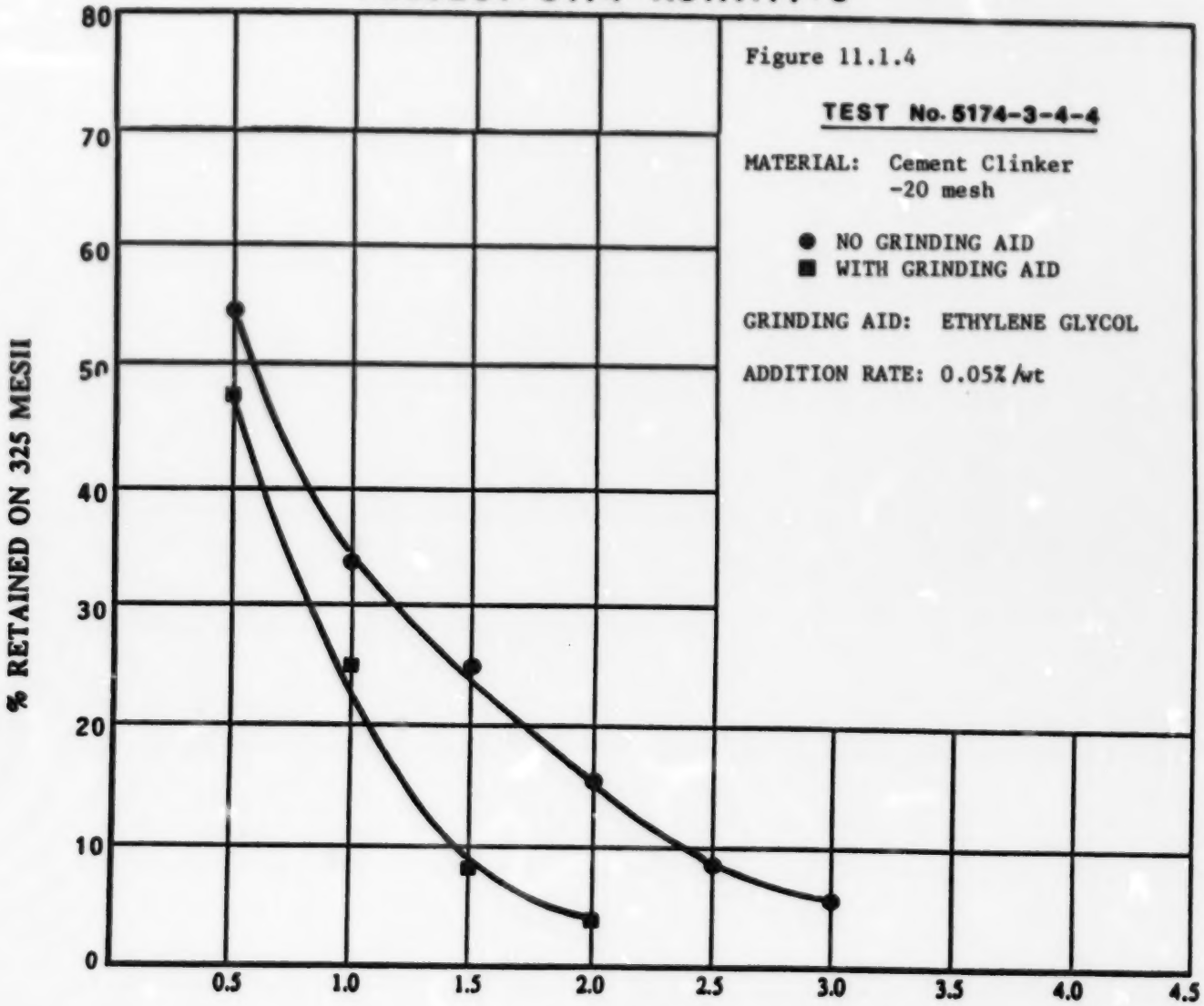


Figure 11.1.4

TEST No. 5174-3-4-4

MATERIAL: Cement Clinker
-20 mesh

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: ETHYLENE GLYCOL

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

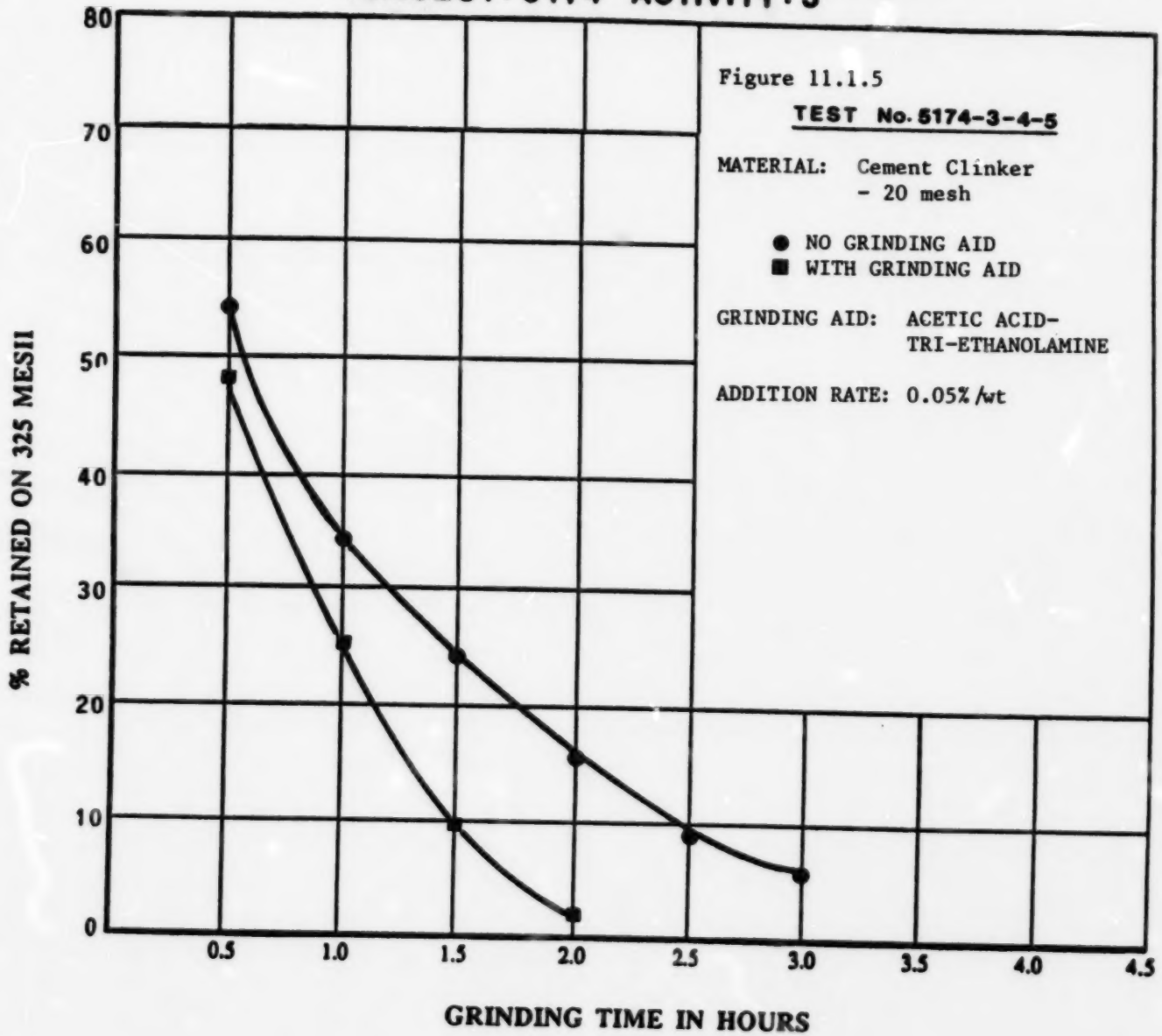


Figure 11.1.5

TEST No. 5174-3-4-5

MATERIAL: Cement Clinker
- 20 mesh

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: ACETIC ACID-
TRI-ETHANOLAMINE

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

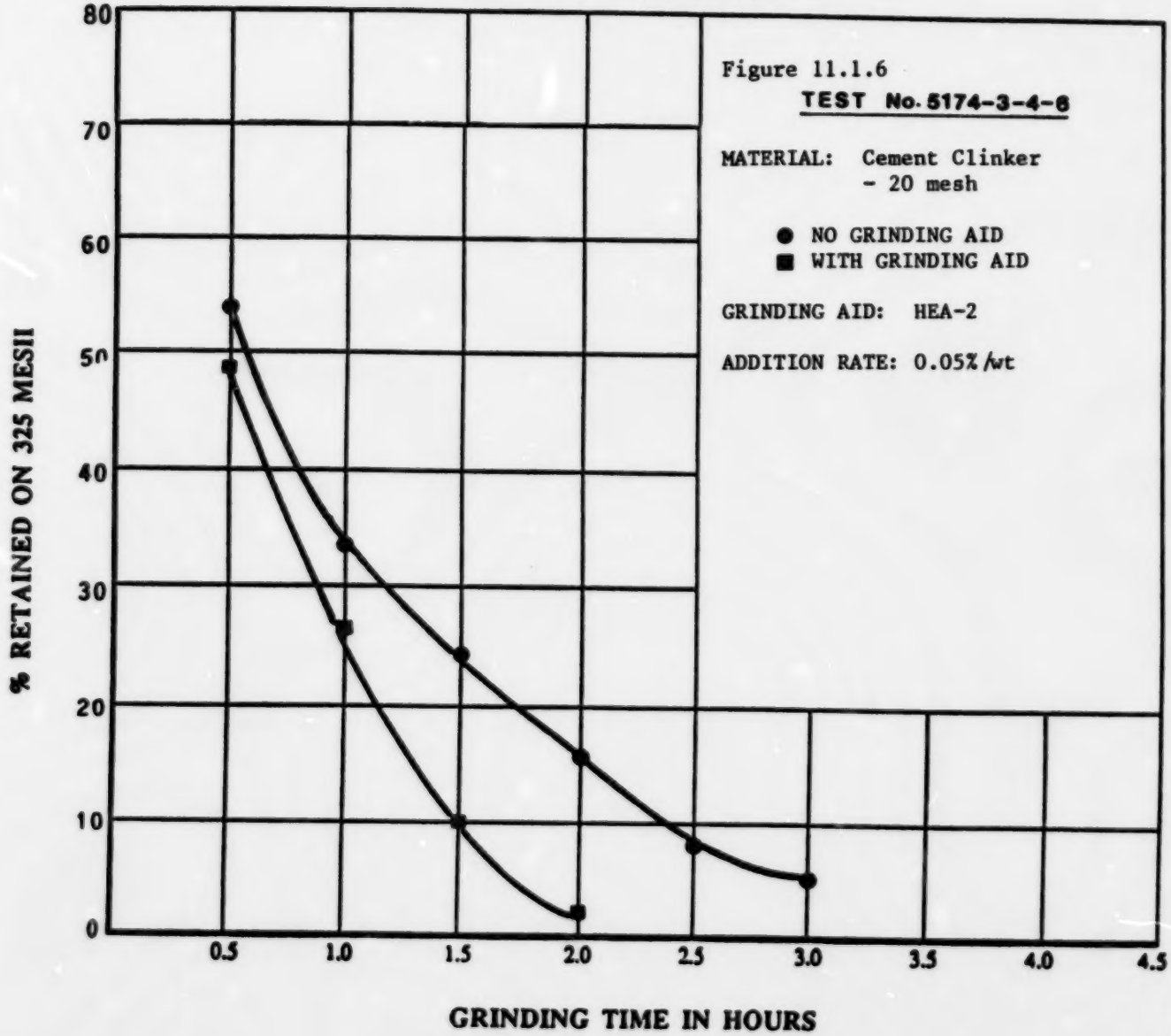


Figure 11.1.6

TEST No. 5174-3-4-6

MATERIAL: Cement Clinker
- 20 mesh

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: HEA-2

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

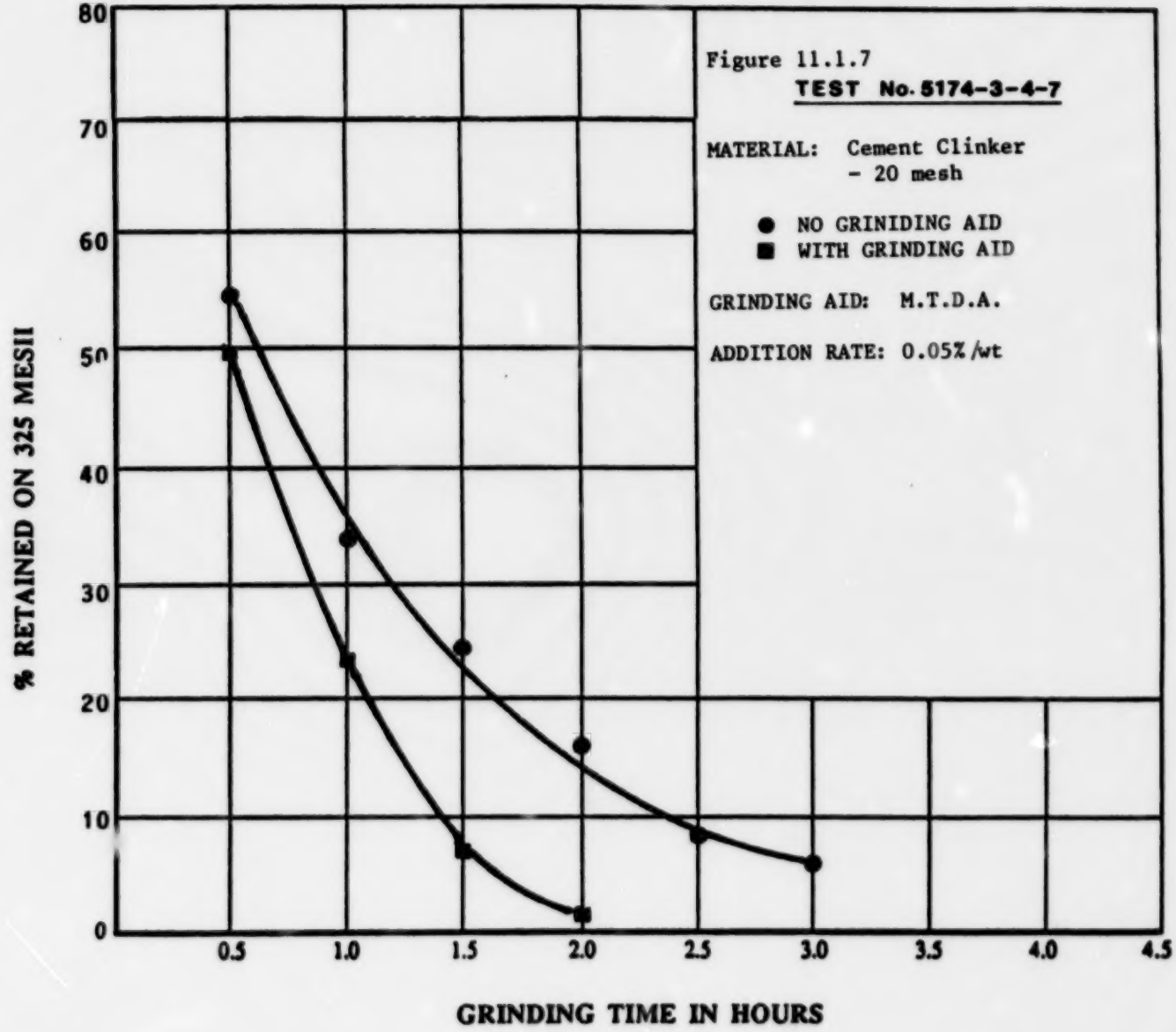


Figure 11.1.7

TEST No. 5174-3-4-7

MATERIAL: Cement Clinker - 20 mesh

● NO GRINIDING AID
■ WITH GRINDING AID

GRINDING AID: M.T.D.A.

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

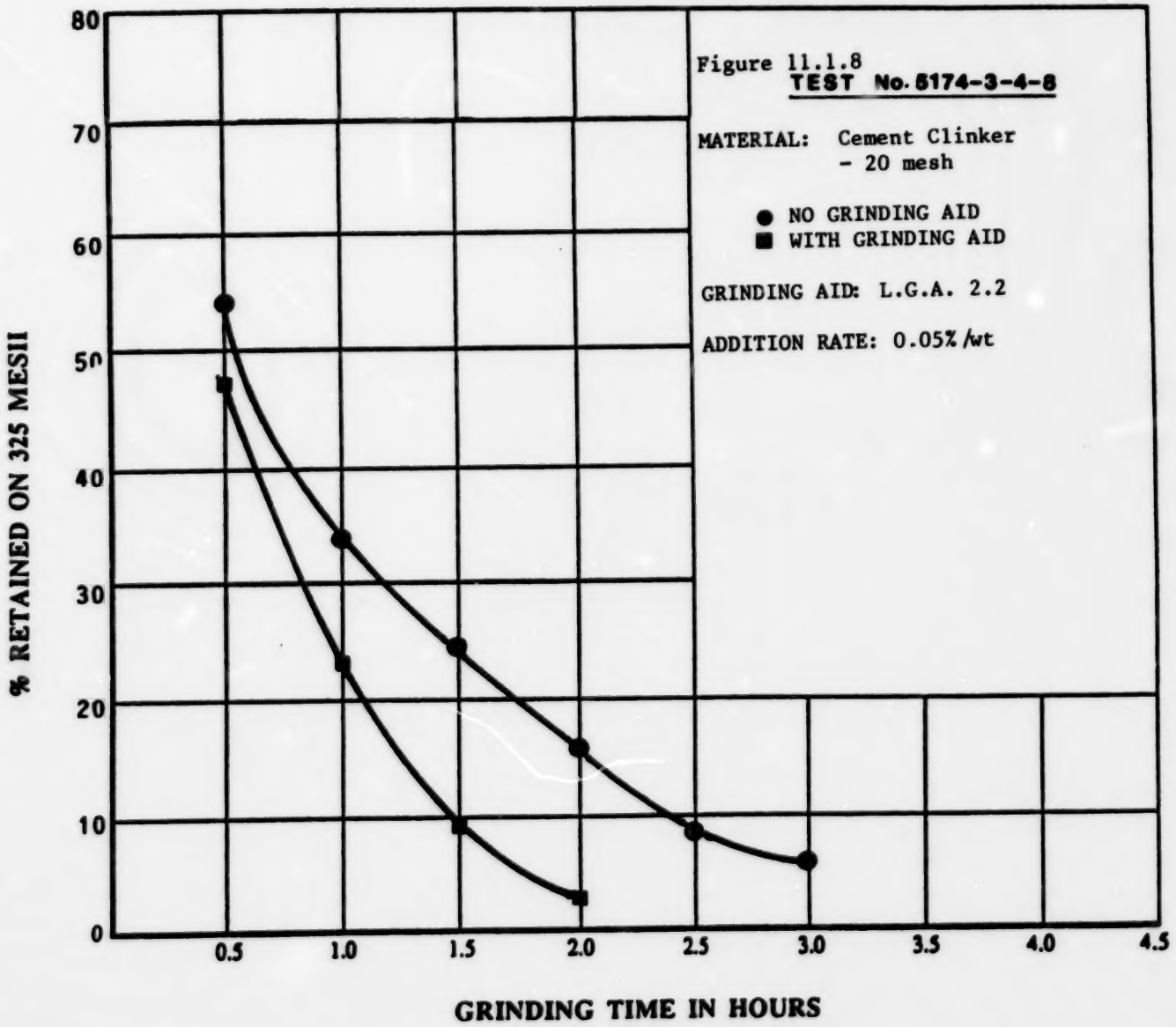


Figure 11.1.8
TEST No. 5174-3-4-8

MATERIAL: Cement Clinker
- 20 mesh

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: L.G.A. 2.2

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

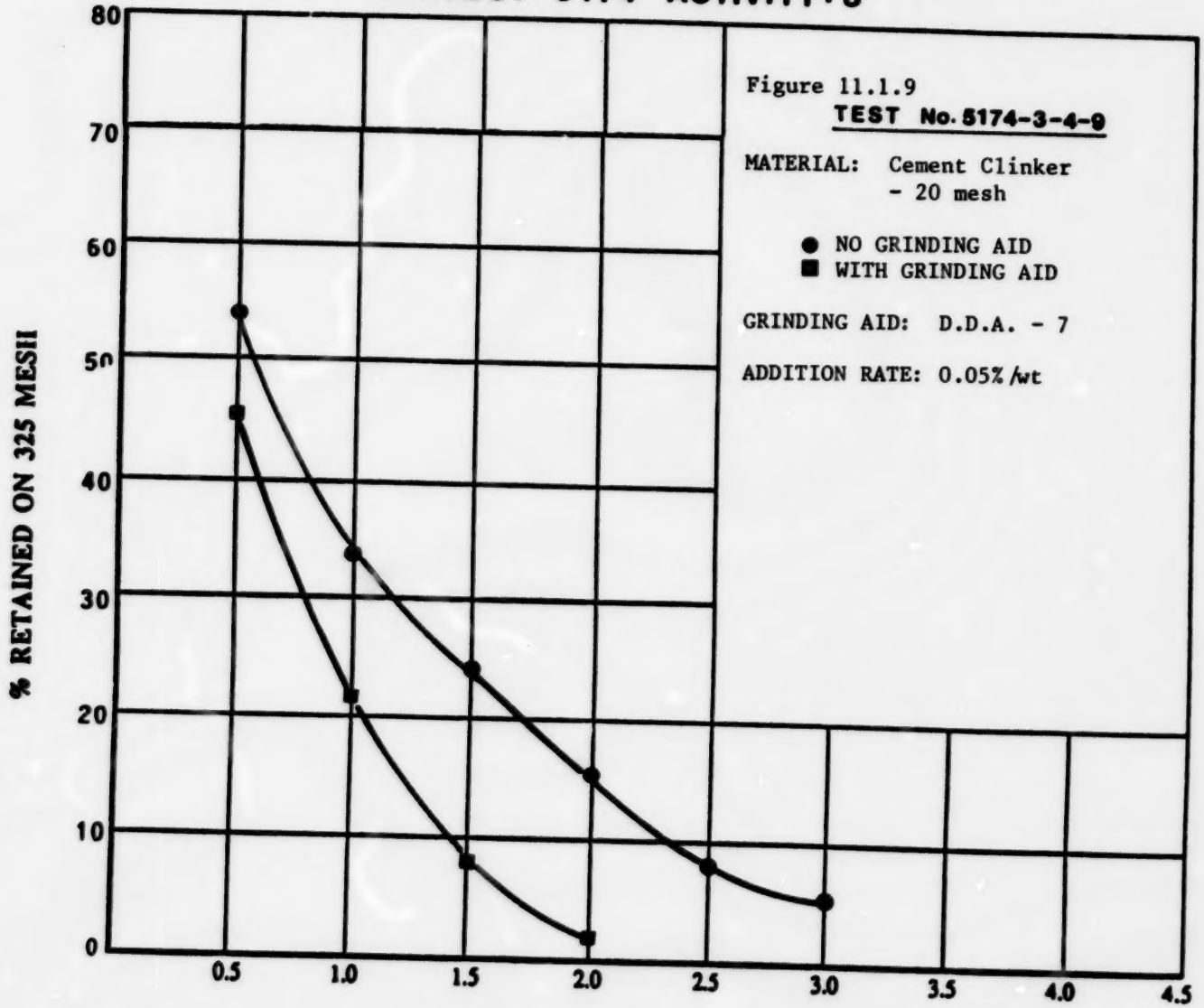


Figure 11.1.9

TEST No. 5174-3-4-9

MATERIAL: Cement Clinker - 20 mesh

● NO GRINDING AID
■ WITH GRINDING AID

GRINDING AID: D.D.A. - 7

ADDITION RATE: 0.05% /wt

DSS-CANADA CEMENT LAFARGE

PROJECT: 5174 ACTIVITY: 3

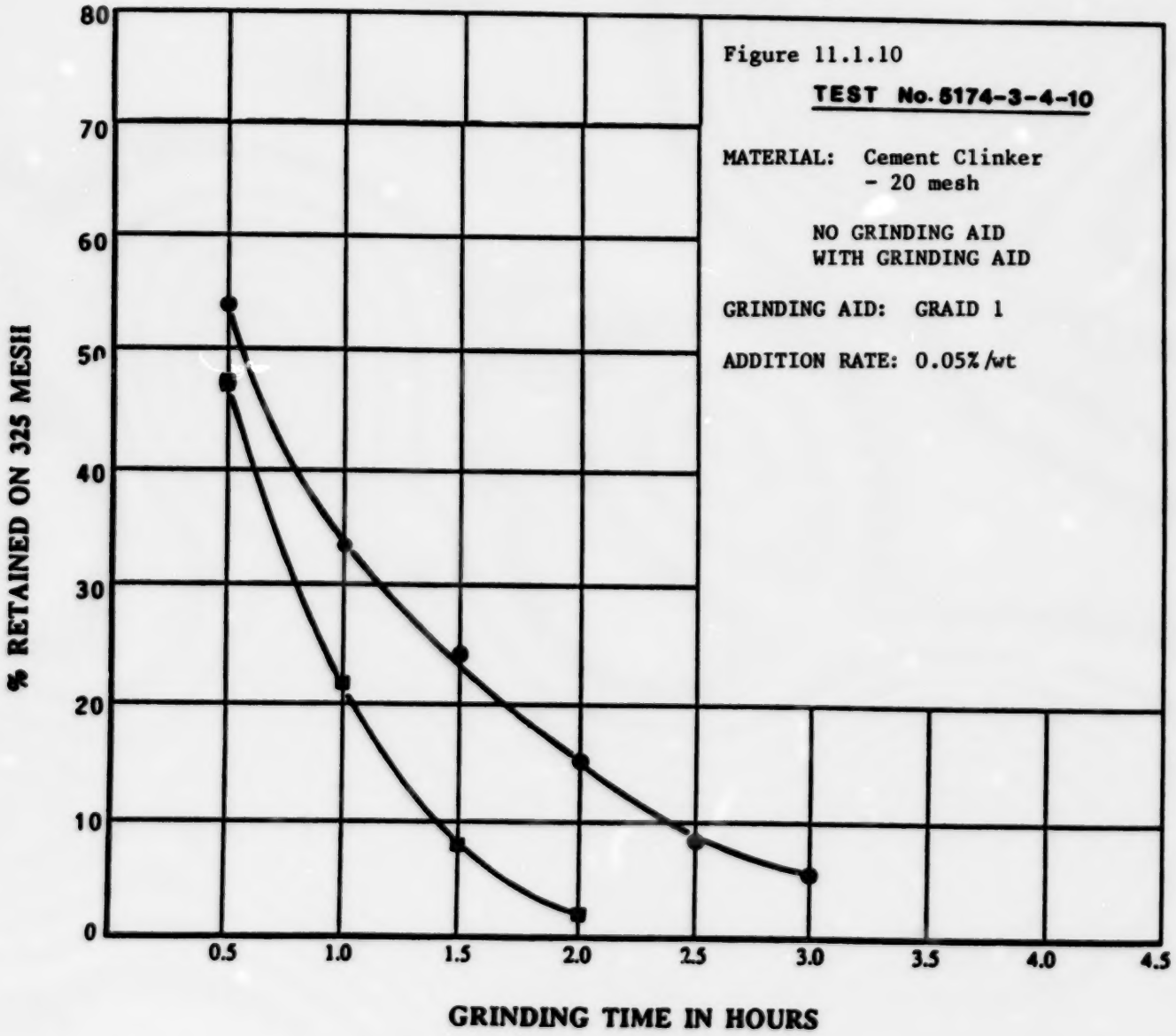


Figure 11.1.10

TEST No. 5174-3-4-10

MATERIAL: Cement Clinker
- 20 mesh

NO GRINDING AID
WITH GRINDING AID

GRINDING AID: GRAID 1

ADDITION RATE: 0.05%/wt

12.0 CONCLUSIONS

The use of chemical additives in dry grinding of non metallic minerals and cement has been the subject of much research and development during the last two decades. The impetus for this research, development and industrial application was the need to conserve energy, particularly after the rapid escalation in the cost of energy in the early seventies.

Research in this field was conducted in most industrialized nations, particularly Europe. Implementation of the use of chemical additives has been most pronounced in the cement industry. Most of the practical data on the benefits of chemical additives has been reported by cement manufacturing world wide.

While research data shows that significant benefits can be realized in the grinding of other non metallic minerals, implementation of chemical additives in these industries appear to be lagging behind the cement industry.

Bench scale grinding tests conducted as part of this project, show conclusively that under the test conditons employed, chemical additives can significantly enhance the effectiveness of the grinding process. In virtually all cases, the improvements can be attributed to the ability of the additive to prevent agglomeration and coating of mill walls and grinding media. Chemical additives tested in this program showed excellent effectiveness in grinding limestone and cement. They were less effective in grinding nepheline syenite and silica. None of the chemical additives tested were effective in grinding talc.

Data obtained from the literature survey shows conclusively that chemical additives when used in the dry grinding of non-metallic minerals and cement can significantly contribute to improved productivity and energy conservation. The primary benefits are as follows:

- Chemical additives or grinding aids are particularly useful in preventing agglomeration of the material ground and in eliminating or reducing the formation of coatings on grinding media and mill walls.

- This allows the grinding process to proceed unimpeded, achieving mill performance as predicted from the Bond Work Index.

- Some chemical additives also act to improve flow through grinding mills or as a dispersant. This can be particularly beneficial in large tube mills and in air classification. Good dispersion of the ground material will improve the efficiency of the classification process..

- The primary benefit of a chemical additive is that it allows the grinding process to operate at its full potential, making efficient use of energy.

- This, in most cases, leads to increased production at equal energy input or, conversely, allows finer grinding of equal amounts of material.

- Improvements in productivity or reduction in energy consumption may range from as low as ten (10) percent to as high as one hundred (100) percent.

- Benefits are most likely to be realized in grinding mills in which serious agglomeration and coating of grinding media and mill walls are experienced and minimal in mills where these problems are not as serious.

- Agglomeration not only affects the grinding process, it also was implicated in air classification. Agglomerates present in the classifier feed will be rejected by the classifier as coarse particles, further compounding grinding problems.

- Optimum benefits are realized when the grinding mill is operated in closed circuit with a high efficiency air classifier. Problems in grinding can be caused by air classifiers that are inefficient, causing large quantitites of product sized material to be returned to the mill. This will negatively effect mill performance, causing overgrinding, poor flow through the mill and create conditions favourable to the formation of agglomerates and mill coatings.

Chemical additives are most effective in fine grinding operations, where agglomeration, media and mill coatings are being experienced. The point or particle size where this phenomena occurs differ from mineral to mineral and even for the same mineral from different deposits.

A specific chemical additive may perform very well on material from one deposit while the same additive does not perform as well on the same material from another deposit. Conversely, another chemical additive may perform well on the second material.

WD1

It is clear that data as reported on the performance of a specific chemical additive cannot be indiscriminately extrapolated to other or similar materials and that careful screening of a number of potential additives is necessary to find the appropriate additive for each specific case.

Implementation of chemical additives in full scale production plants must be done with care. The use of chemical additives may cause a significant change in the operational characteristics of a mill. Mill residence time may change because of improved material flow, resulting in increased circulating load. This can be corrected by modifying the discharge grates in the mill.

Of particular importance in the selection of a chemical additive is to ascertain the effect chemical additives may have on the performance of the products in industrial applications.

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   Patent Assignee: (TERO) TEROSON WERKE GMBH
   Author (inventor): SCHENKER A; SCHUTZ J; SCHNEIDER J

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   PATENT: United States US 3329517 DATE: 670704
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   PATENT: Japan Kokai Tokkyo Koho: JP 81109852 A2
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